

# Quality Assurance Manual

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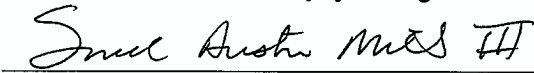
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## **SOPs AND POLICIES REFERRED TO IN THE QA MANUAL**

<b>SOP/Policy Reference</b>	<b>Title</b>
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-003	Management of Change Procedure
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-005	Calibration Curves (General)
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-L-P-001	Record Retention
CW-F-P-002	Authorization Matrix
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-004	Controlled Purchases Policy

## SECTION 3

### INTRODUCTION (NELAC 5.1 - 5.3)

#### 3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica Knoxville's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. Each TestAmerica laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (1999). In addition, the policies and procedures outlined in this manual are compliant with the various accreditation and certification programs listed in Appendix 6. The relevant NELAC section is included in the heading of each QAM section.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*, EPA, March 1979.
- EPA SW-846, *Test Methods for the Evaluation of Solid Waste*, 3<sup>rd</sup> Edition, September 1986; Update I, July 1992; Update II, September 1994; and Update III, December 1996.
- Federal Register, 40 CFR Parts 136, 141, and 261.
- APHA, *Standard Methods for the Examination of Water and Wastewater*, 18<sup>th</sup> Edition, 19<sup>th</sup>, 20<sup>th</sup> and 21<sup>st</sup> Edition.
- U.S. Department of Energy Order 414.1B, *Quality Assurance*, Approved April 29, 2004, and/or 414.1C, *Quality Assurance*, Approved June 17, 2005; and U.S. Department of Energy Order 450.1, *Environmental Management Systems*, Approved January, 15, 2003.
- U.S. Department of Energy, *Quality Systems for Analytical Services*, Revision 2.3, October 2007.
- U.S. Department of Energy Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- U.S. Department of Energy, *Quality Systems for Analytical Services*, Revision 2.2, October 2006.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 3, January 2006.
- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan(QAPP)*, Version 4.0.02, May 2006.

#### 3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by TestAmerica Knoxville conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 5 for the Glossary/Acronyms.

### **3.3 SCOPE / FIELDS OF TESTING**

TestAmerica Knoxville analyzes hundreds of environmental and industrial samples every month. Sample matrices vary among air (source and ambient), drinking water, effluent water, groundwater, soils, sediment, biological, hazardous waste, and sludge. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, and physical parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested water, air, industrial waste, and soil methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Table 20-1. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by TestAmerica Knoxville shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

### **3.4 MANAGEMENT OF THE MANUAL**

#### **3.4.1 Review Process**

The manual is reviewed annually by the QA Manager and laboratory personnel to assure that it reflects current practices and meets the requirements of TestAmerica Knoxville's clients and regulators. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. The updates will be reviewed by the QA Manager, Laboratory Director, Technical Director(s), relevant operational staff and Corporate Quality Assurance (if a change is made to the Corporate template) and then formally incorporated into the document in periodic updates. The QAM is based on a Corporate QAM Template that is prepared and approved by the Chief Operating Officers (COOs) and Corporate Quality Assurance. This template is reviewed annually by the COOs, Corporate Quality, and each laboratory. Necessary changes are coordinated by the Vice President of Quality and Environmental Health & Safety (EHS) and distributed to each laboratory for inclusion in the laboratory specific QA Manuals.

Policies in the QAM that require immediate attention must be addressed through the use of Corporate QA/QC Policy Memoranda or by revision of the appropriate section of the QAM.

QA/QC Policy Memoranda are published from time to time to facilitate immediate changes to QA/QC Policy. QA/QC Policy Memoranda supersede the QAM and all other SOPs (refer to Section 5.3). All policy memoranda are dated, archived and distributed by their placement into the front of the QAM between the signature page and Section 2. At a minimum, each policy memorandum is approved by the same authorized signatories as shown on the cover page of the QA Manual. In addition, Corporate QA/QC Policy Memoranda are signed by the COOs and VP of Quality and EHS. The QA/QC Policy Memoranda are incorporated into the QAM during the periodic updates. Policy memorandum may also include an expiration date if appropriate. An example format can be found in Figure 3-1. A similar procedure is followed for local laboratory changes.

Laboratory-specific QAM changes are initiated through the TestAmerica Knoxville Quality Assurance Manager. Each revision to the QA Manual is approved by the same authorized signatories as shown on the cover page of the QA Manual. Changes to the Laboratory Specific QAM must also be reviewed and approved by Ohio EPA VAP prior to implementation.

#### **3.4.2     Control**

This manual is considered confidential within TestAmerica and must not be altered in any manner by other than a duly appointed representative from TestAmerica. If the document has been provided to external users or regulators, it is for the exclusive purpose of reviewing TestAmerica Knoxville's quality systems and shall not be used in any other way without the written permission of an appointed representative of TestAmerica. The procedure for control of distribution is incorporated by reference to KNOX-QA-0011 Document Control and Distribution.

The order of precedence in the event of a conflict between policies is outlined in Section 5.3 of this Quality Assurance Manual.

**Figure 3-1.**

**Example - Format for a QA/QC Policy Memorandum**

**Corporate (or Laboratory) QA/QC Policy Memorandum # \_\_\_\_\_**

Effective Date: \_\_\_\_\_ Expiration Date: When Appropriate QAM Section is Revised

**Corporate:** *(Only needed for Corporate Memorandum – Delete if Laboratory)*

_____ COO - West	_____ Date	_____ Vice-President, QA and EHS	_____ Date
---------------------	---------------	-------------------------------------	---------------

_____ COO - East	_____ Date
---------------------	---------------

**Local:**

_____ Technical Director Approval	_____ Date	_____ Technical Director Approval	_____ Date
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_____ Quality Assurance Approval	_____ Date	_____ Laboratory Director Approval	_____ Date
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**1. Purpose**

**2. Procedure**

**3. Attachments**

**4. References/Cross References**

## SECTION 4

### ORGANIZATION AND MANAGEMENT (NELAC 5.4.1)

#### 4.1 OVERVIEW

TestAmerica Knoxville is part of a national network of laboratories known as TestAmerica. This Quality Assurance Manual (QAM) is applicable to the TestAmerica Knoxville laboratory only.

TestAmerica Knoxville  
5815 Middlebrook Pike  
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EPA ID Code: TN00009

The Corporate organization chart can be found in Figure 4-1 and the laboratory's organization chart can be found in Appendix 2. The locations of other TestAmerica labs are as follows:

TestAmerica Anchorage  
TestAmerica Austin  
TestAmerica Buffalo  
TestAmerica Burlington  
TestAmerica Cedar Falls  
TestAmerica Chicago  
TestAmerica Connecticut  
TestAmerica Corpus Christi  
TestAmerica Dayton  
TestAmerica Denver  
TestAmerica Edison  
TestAmerica Honolulu  
TestAmerica Houston  
TestAmerica Irvine  
TestAmerica King of Prussia  
TestAmerica Los Angeles  
TestAmerica Mobile  
TestAmerica Morgan Hill  
TestAmerica Nashville  
TestAmerica North Canton  
TestAmerica Ontario  
TestAmerica Orlando  
TestAmerica Pensacola  
TestAmerica Phoenix  
TestAmerica Pittsburgh  
TestAmerica Portland  
TestAmerica Richland  
TestAmerica San Francisco  
TestAmerica Savannah  
TestAmerica Seattle

TestAmerica Spokane  
TestAmerica St. Louis  
TestAmerica Tacoma  
TestAmerica Tallahassee  
TestAmerica Tampa  
TestAmerica Valparaiso  
TestAmerica Watertown  
TestAmerica West Sacramento  
TestAmerica Westfield

## **4.2 ROLES AND RESPONSIBILITIES**

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions define each role in its relationship to the Quality Assurance Program. More detailed laboratory job descriptions are maintained by management and human resources on the company intranet.

### **4.2.1 Quality Assurance Program**

The responsibility for quality lies with every employee of TestAmerica Knoxville. All employees have access to the QAM and are responsible for knowing the content of this manual and upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs.

### **4.2.2 Chairman/Chief Executive Officer (CEO)**

The Chairman/CEO is the Chairman of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. Together with the President/CEO of the Analytical Division, the Chairman/CEO establishes the overall quality standard and data integrity program for the company, providing the necessary leadership and resources to assure that the standard and integrity program are met.

### **4.2.3 President/Chief Executive Officer (CEO)**

The President/CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. Together with the Chairman/CEO, the President/CEO establishes the overall quality standard and data integrity program for the Analytical Division, providing the necessary leadership and resources to assure that the standard and integrity program are met.

### **4.2.4 Chief Operating Officer (COO) – East and West**

The COOs serve as the ranking executives for all respective analytical laboratory operational functions and report to the President/CEO of the Analytical Division. They are responsible for the daily management of all analytical laboratories, long-term planning and development of technical policies and management plans. They ensure the attainment of corporate objectives through the selection, development, motivation, and evaluation of top management personnel.



The COOs approve all operating budgets and capital expenditures. The COOs sign-off on the final QAM template that contains company policies for implementing the Quality Program.

#### **4.2.5 General Manager (GM)**

Each GM reports directly to a COO. Each GM has full responsibility for the overall administrative and operational management of their respective laboratories. The GM's responsibilities include allocation of personnel and resources, long-term planning, setting goals, and achieving the financial, business, and quality objectives of TestAmerica. The GM ensures timely compliance with corporate management directives, policies, and management systems reviews. The GM is also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

#### **4.2.6 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)**

The Vice President of QA/EHS reports directly to the Chairman/CEO. With the aid of the Analytical Division and Non-Analytical Division Senior Management Teams, Laboratory Director/Managers, Quality Directors, EHS Directors, QA Managers and EHS Coordinators, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and Environmental, Health and Safety Program within TestAmerica. Additional responsibilities include:

- Review of QA/QC aspects of Corporate SOPs, national projects and expansions or changes in services.
- Coordination/preparation of the Corporate QAM Template that is used by each laboratory to prepare its own laboratory-specific QAM.
- Maintenance of Corporate Policies, Quality Memorandums and SOPs. Maintenance of data investigation records that are reported to Corporate Management.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation of a monthly report that includes quality metrics across the Analytical Division and a summary of any quality related initiatives and issues.
- With the assistance of the Corporate Senior Management Teams and the EHS Directors, development and implementation of the TestAmerica Environmental, Health and Safety Program.

#### **4.2.7 Quality Directors (Corporate)**

The Quality Directors report to the VP-QA/EHS. Together with the VP-QA/EHS, the Quality Directors have the responsibility for the establishment, general overview and maintenance of the Analytical Division's Quality Assurance Program within TestAmerica. The Quality Directors are responsible for:

- Oversight of the QA/QC programs within each laboratory. This includes a final review of each laboratory-specific QAM and receipt of each laboratory's QA monthly report.
- Review of QA/QC aspects of national projects.
- Assistance with certification activities.

#### **4.2.8 Ethics and Compliance Officers (ECOs)**

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – VP-QA/EHS and VP-Client and Technical Services. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the CEOs, COOs, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

#### **4.2.9 Vice President of Client and Technical Services**

The Vice President (VP) of Client and Technical Services is responsible for offerings to clients including risk management, technical assistance, legal compliance and contract administration. The VP of Client and Technical Services provides support and direction to the Managers of these areas, and supports the COOs in decisions regarding long term planning, resource allocation and capital expenditures.

#### **4.2.10 Director of Technical Services**

The Director of Technical Services is responsible for establishing, implementing and communicating TestAmerica's Analytical Division's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

#### **4.2.11 Chief Information Officer (CIO)**

The CIO is responsible for establishing, implementing and communicating TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

#### **4.2.12 Environmental Health and Safety Directors (EHSDs) (Corporate)**

The EHSDs report directly to the VP-QA/EHS. The EHSDs are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

#### **4.2.13     Laboratory Director**

TestAmerica Knoxville's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the laboratory and reports directly to the General Manager. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Implementing and ensuring adherence to the Quality System as described in this QA Manual and in the supporting laboratory policies and procedures.
- Providing one or more technical directors for the appropriate fields of testing. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensuring that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensuring that this training has been documented.
- Ensuring that personnel are free from any undue pressures, including inappropriate commercial or financial concerns which may adversely affect the quality of their work.
- Annually assessing the effectiveness of the Quality System within the operation.
- Maintaining adequate trained staff to supervise and perform the work of the laboratory.
- Ensuring that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Having signature authority for the QAM, laboratory policies, SOPs, and contracts.

#### **4.2.14     Quality Assurance (QA) Manager**

The QA Manager is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. Responsibilities include:

- Reporting directly to the Laboratory Director and to the Quality Director to maintain independence of QA oversight.
- Maintaining, approving, communicating and implementing the QA Manual.
- Having joint signature authority, with the Laboratory Director and Technical Director for approval of quality documents.
- Directing controlled distribution of laboratory quality documents.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Reviewing and approving documentation of analyst training records.

- Serving as a focal point for QA and QC issues, reviewing corrective actions and recommending resolution for recurring nonconformances within the laboratory.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 13.
- Assisting the laboratory in maintaining regulatory analytical compliance, including maintaining certifications.
- Having a general knowledge of the analytical test methods for which data audit/review is performed.
- Monitoring data quality measures via statistical methods to verify that the laboratory routinely meets stated quality goals.
- Scheduling proficiency test samples.
- Performing annual quality systems audits, and periodic data audits.
- Hosting external audits conducted by outside agencies.
- Approving quality control reference data changes in the LIMS.
- Preparing monthly QA Reports to management providing Quality System Metrics.
- Having the final authority to accept or reject data and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data.

#### **4.2.15     Technical Director**

The Technical Director is responsible for the technical operation of the laboratory and reports directly to the Laboratory Director. Specific responsibilities include, but are not limited to:

- Monitoring standards of performance in quality control and quality assurance.
- Monitoring the validity of the analyses performed and the data generated in the laboratory to assure reliable data.
- Exercising day to day supervision of laboratory operations and data reporting.
- Coordinating the development and implementation of SOPs.
- Having joint signature authority for QAM, SOPs, and training records.
- Performing technical training in area(s) of expertise.
- Interfacing with management on technical needs and solving day-to-day technical issues.
- Determining qualifications required for technical positions and evaluating job candidates against those requirements.
- Investigating technical issues related to projects as directed by QA.
- Evaluating new methods, technical proposals, and statements of work.
- Certifying technical laboratory personnel based on education and background to ensure that staff have demonstrated capability in the activities for which they are responsible.
- Meeting the requirements specified in the Section 4.1.1.1 of the NELAC standards.

#### **4.2.16     Operations Manager**

The Operations Manager reports directly to the Laboratory Director and supervises the daily activities of the analytical groups. Responsibilities include, but are not limited to:

- Scheduling analytical operations.
- Supervising QC activities performed as a part of routine analytical operations.
- Implementing data review procedures.
- Supervising the preparation and maintenance of laboratory records.
- Supervising maintenance of instruments and scheduling of repairs.
- Supervising the daily activities of the Report Production Group.
- Working with the Project Managers and Group/Team Leaders to assure the requirements of projects are met in a timely manner.

#### **4.2.17     Report Production Staff**

Responsibilities include, but are not limited to:

- Accurately generating and compiling analytical reports and associated deliverables for delivery to the client.
- Producing deliverables that meet NELAC requirements, as required.

#### **4.2.18     Environmental Health and Safety Coordinator and Radiation Safety Officer**

The Environmental Health and Safety Coordinator reports directly to the Laboratory Director. Responsibilities include, but are not limited to:

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan and the Corporate Safety Manual.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.
- Conducting ongoing, necessary safety training and conduct new employee safety orientation.
- Assisting in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administering dispersal of all Material Safety Data Sheet (MSDS) information.
- Performing regular chemical hygiene and housekeeping instruction.
- Giving instruction on proper labeling and practice; Serving as chairman of the laboratory safety committee.

- Overseeing the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervising and scheduling fire drills and emergency evacuation drills.

As Radiation Safety Officer, responsibilities include but are not limited to:

- ensuring that all uses of radioactive materials and radiation sources are conducted safely, in accordance with the conditions of the license and applicable regulations, and result in exposures to personnel which are as low as reasonably achievable (ALARA).
- apprising and informing management of the radiation safety status of the facility and for keeping them aware of their responsibility in maintaining an adequate radiation protection program.

#### **4.2.19 Group (Area) Leader, Team Leader or Supervisor**

Supervisors report directly to the Operations Manager or the Laboratory Director. Responsibilities include, but are not limited to:

- Supervising daily activities of analyses within the group ensuring that the analysts adhere to the appropriate SOPs and the QA Manual.
- Supervising QC activities performed as a part of routine analytical operations.
- Implementing data review procedures.
- Supervising the preparation and maintenance of laboratory records.
- Evaluating instrument performance and supervising the calibration, preventive maintenance, and scheduling of repairs.
- Overseeing or performing review and approval of all analytical data.
- Reporting nonconformances to the appropriate managers.
- Generating and reviewing SOPs for their section.

#### **4.2.20 Laboratory Analysts**

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. Responsibilities include, but are not limited to:

- Performing analytical methods and data recording in accordance with documented procedures.
- Performing and documenting calibration and preventive maintenance.
- Performing data processing and data review procedures.
- Reporting nonconformances to the Supervisor/Manager and QA Manager.
- Ensuring sample and data integrity by adhering to internal chain-of-custody procedures.

#### **4.2.21 Sample Receiving Group Leader**



The Sample Receiving Group Leader reports to the Customer Service Manager. Responsibilities include, but are not limited to:

- Ensuring implementation of proper sample receipt procedures, including maintenance of chain-of-custody.
- Reporting variances associated with condition-upon-receipt of samples.
- Logging samples into the LIMS.
- Ensuring that all samples are stored in the proper environment.
- Assisting Environmental Health and Safety staff with sample disposal.

#### **4.2.22     Customer Service Manager**

The Customer Service Manager reports directly to the Laboratory Director and serves as the interface between the laboratory's technical departments and the laboratory's clients. The staff consists of the Project Management team. Responsibilities include, but are not limited to:

- Supervising the daily activities of the Project Management and Sample Control groups.
- Working with the Operations Manager and/or Group/Team Leaders to ensure the requirements of projects are met in a timely manner.
- Having signature authority for laboratory reports.
- Defining customer requirements through project definition.
- Assessing and assuring customer satisfaction.
- Providing feedback to management on changing customer needs.
- Bringing together resources necessary to ensure customer satisfaction.

#### **4.2.23     Project Manager**

The Project Manager reports directly to the Customer Service Manager. Responsibilities include, but are not limited to:

- Monitoring analytical and QA project requirements for a specified project.
- Acting as a liaison between the client and the laboratory staff.
- Preparing Quality Assurance Summary (QAS) or equivalent summary form and communicates project-specific requirements to all parties involved.
- Assisting the laboratory staff with interpretation of work plans, contracts, and QAPP requirements.
- Reviewing project data packages for completeness and compliance to client needs.
- Having signature authority for final report.
- Keeping the laboratory and client informed of project status.
- Together with the QA Manager, approving customer requested variances to methods and to standard laboratory protocols.

- Monitoring, reviewing, and evaluating the progress and performance of projects.
- Reporting client inquiries involving data quality issues or data acceptability to the facility QA Manager and to the operations staff.
- Preparing reissue requests for project data.

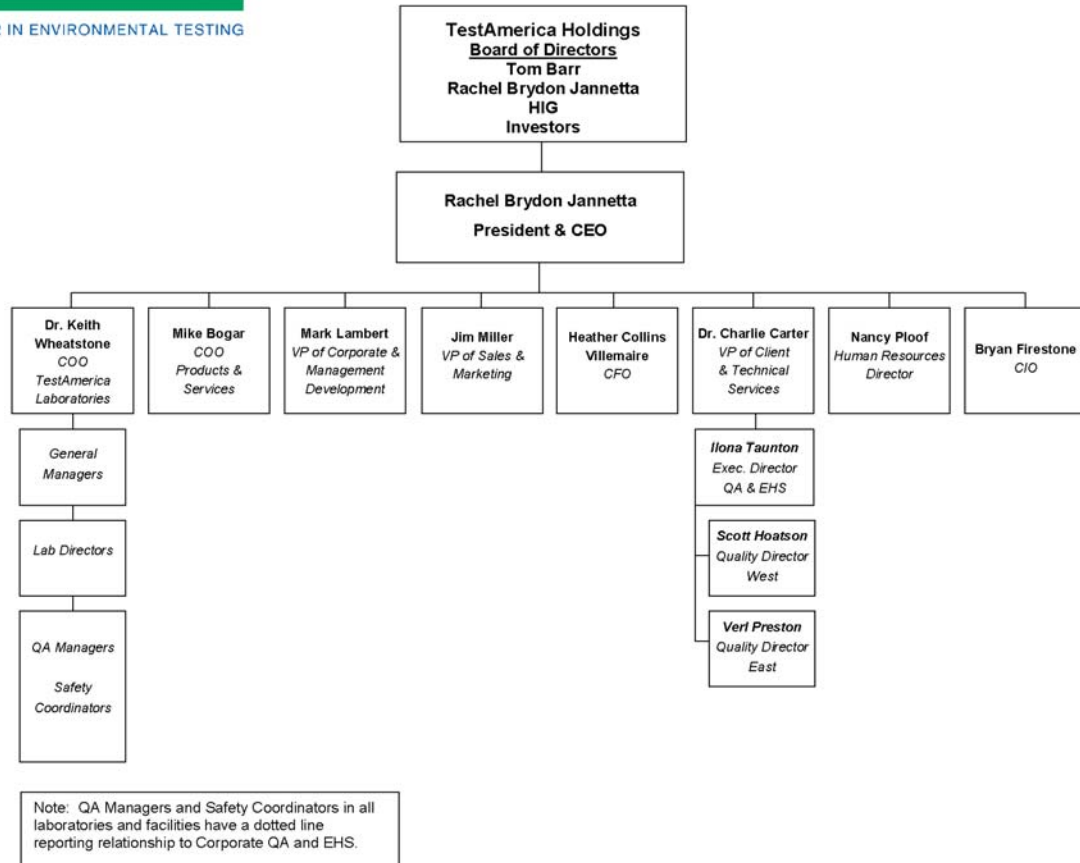
#### **4.3      DEPUTIES**

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Laboratory Director: Tom Yoder	Robyn Wagner
QA Manager: Christopher Rigell	Vanhseng (Mo) Khounlavong
Technical Director: Robyn Wagner	Christopher Rigell
Technical Director: Snell Mills	Melissa Davidson
Customer Service Manager: John Reynolds	Linda McWhirter
EHS Coordinator: Lewis Osborne	Scot Goss
Radiation Safety Officer (RSO): Lewis Osborne	Scot Goss

Figure 4-1.

Corporate Organization Chart



## **SECTION 5**

### **QUALITY SYSTEM (NELAC 5.4.2)**

#### **5.1 QUALITY POLICY STATEMENT**

The management of TestAmerica and TestAmerica Knoxville are committed to providing data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols described in this manual.

In all aspects of the laboratory and business operations, management is dedicated in maintaining the highest ethical standards. An Ethics Policy sign-off can be viewed in Appendix 1. Training on ethical and legal responsibilities is provided annually and each employee signs off annually on the policy as a condition of employment.

It is TestAmerica's Policy to continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. The company recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.

TestAmerica Knoxville strives to provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at TestAmerica Knoxville plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

#### **5.2 ETHICS AND DATA INTEGRITY**

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The 7 elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Policy No. CA-L-P-001) and employee ethics statements (Appendix 1).
- An Ethics and Compliance Officer (ECO).
- A training program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct (SOP No. CA-L-S-001).
- Procedures and guidance for recalling data if necessary (SOP No. CA-L-S-001).
- An effective external and internal monitoring system that includes procedures for internal audits (Section 16).

As an American Council of Independent Laboratories (ACIL) member, all TestAmerica laboratories adhere to the following ACIL Code of Ethics:

- Produce results which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the ethical and quality standards of our industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

### **5.3 QUALITY SYSTEM SUPPORTING DOCUMENTATION**

The laboratory's Quality System is communicated through a variety of documents prepared by the laboratory and company management:

- Quality Assurance Manual (QAM) Template
- Quality Assurance Manual – Each laboratory has a lab specific quality assurance manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical.
- Corporate TestAmerica QA/QC Policy Memorandums (Refer to Section 3.4).
- Laboratory QA/QC Policy Memorandums (Refer to Section 3.4).

#### **5.3.1 Order of Precedence**

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- TestAmerica QA/QC Policy Memorandum - Corporate
- Laboratory QA/QC Policy Memorandum
- Quality Assurance Manual
- Corporate SOPs and Policies
- Laboratory SOPs and Policies

- Other (Work Instructions (WI), memos, flow charts, etc.)

#### **5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA**

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term "*analytical quality control*". QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

##### **5.4.1 Precision**

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples. The calculation of precision is described in Section 25.

##### **5.4.2 Accuracy**

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery. The calculation of accuracy is described in Section 25.

#### **5.4.3      Representativeness**

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

#### **5.4.4      Comparability**

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories, and by the degree to which approval from the US EPA or other pertinent regulatory agencies is obtained for any procedure for which significant modifications have been made.

#### **5.4.5      Completeness**

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

#### **5.4.6      Selectivity**

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..



#### **5.4.7      Sensitivity**

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit, Estimated Detection Limit, or Limit of Detection) or quantified (Reporting Limit, Minimum Level, or Limit of Quantitation).

### **5.5            CRITERIA FOR QUALITY INDICATORS**

The laboratory can prepare upon request a Quality Control Limit Summary from the LIMS that summarize the precision and accuracy acceptability limits for analyses performed at TestAmerica Knoxville. This summary includes an effective date, is updated each time new limits are generated and is located in the LIMS. Current limits are controlled through the LIMS. The limits in effect for a given date are archived in the LIMS with the associated sample data. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, TestAmerica Knoxville has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in Section 25.

### **5.6            STATISTICAL QUALITY CONTROL**

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. TestAmerica Knoxville routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Director/Group Supervisor and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. These limits are maintained in the LIMS as part of the analytical historical record. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

#### **5.6.1      QC Charts**

The generation and use of QC Charts (Control Charts) are described in the laboratory SOP KNOX-QA-0004.

## **5.7      QUALITY SYSTEM METRICS**

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 17). These metrics are used to drive continuous improvement in the laboratory's Quality System.

## SECTION 6

### DOCUMENT CONTROL (NELAC 5.4.3)

#### 6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled at each laboratory Facility:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

The Corporate staff posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These are collectively termed "Official Documents" and encompass the Policies and Procedures that all facilities are required to employ. These official documents are only considered controlled when they are read on the company intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving official documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving and in KNOX-QA-0011, Document Control and Distribution.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and nonconformance memos (NCMs). Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports. Discussion on records control is described in Section 15.

The maintenance of purchasing data is discussed in Section 9.

The maintenance of sales and marketing contracts is discussed in Section 7.

#### 6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a control system for each document include a unique name and number, the number of pages of the item, the effective date, revision number and the

laboratory's name. The QA staff is responsible for the maintenance of the system and maintains the items in the QA office archives or the QA electronic archives.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, an employee submits a draft to the QA Department by way of the group leader/supervisor/Technical Director. Upon approval, QA personnel add the identifying version information to the document and retain the official document on file. The official document is provided as needed to those using it. Controlled documents shall be available at all locations where the operational activity described in the document is performed (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years and revised as appropriate. Policies and Procedures used in support of work done for the Department of Defense (DOD), and the TestAmerica Knoxville Quality Assurance Manual must be reviewed at a one year frequency. Changes to documents occur when a procedural change warrants a revision of the document.

### **6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY**

For changes to the QA Manual, requests for change or revision may be made to the TestAmerica QA Manager. Changes affecting the text of the Corporate Template QA Manual are forwarded to the Corporate QA Director for review and approval. If the proposed changes are accepted, they are incorporated into the next revision of the appropriate section of the QA Manual. Each revision to the QA Manual is approved by the same authorized signatories as shown on the cover page of the QA Manual. Uncontrolled copies must not be used within the laboratory. Previous revisions are archived by the QA department.

For changes to SOPs, refer to SOP No. KNOX-QA-0011, Preparation and Management of Standard Operating Procedures.

Forms, worksheets, work instructions and information are organized by form number and revision number on the QAQC shared directory on a local area network server.

#### **6.4      OBSOLETE DOCUMENTS**

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived as described in Section 15.

## SECTION 7

### REVIEW OF WORK REQUEST

#### 7.1 OVERVIEW

TestAmerica Knoxville has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is TestAmerica's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and TestAmerica's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the lab's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and TestAmerica's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the

contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The review process is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

## **7.2 REVIEW SEQUENCE AND KEY PERSONNEL**

The contract review process is outlined in SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements. Appropriate personnel may include:

- Legal & Contracts Director
- General Manager
- The Laboratory Project Manager or Customer Service Manager
- The Laboratory Operations Manager
- Laboratory and/or Corporate Technical Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs.

The National Account Director, Legal Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts. The Project Manager also maintains a copy.

## **7.3 DOCUMENTATION**

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. This information is archived in the project files.



The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Regional Account Manager. A copy of the contract and formal quote will be filed with the laboratory PM and/or the Lab Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client. This information is archived in the project file or in the lot folder as appropriate.

### **7.3.1 Project-Specific Quality Planning**

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, TestAmerica Knoxville assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the direct client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Project specific instructions are communicated to the laboratory through the use of the Client Requirements Checklist in the LIMS or by a Quality Assurance Summary. Further details regarding the communication and documentation of project requirements are found in SOP KNOX-AD-0003, Documenting Project Requirements.

TestAmerica strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

## SECTION 8

### SUBCONTRACTING OF TESTS (NELAC 5.4.5)

#### 8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the corporate network. The phrase “work sharing” refers to internal transfers of samples between company laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When we must outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the SOP on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accredited work where required.

For DOD projects the subcontractor laboratories used must have an established and documented laboratory quality system that complies with DoD QSM requirements. The subcontractor laboratories are evaluated following the procedures outlined below and as seen in Figure 8-1.

The QSM has 5 specific requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.
2. Subcontractor laboratories must be approved by the specific DoD Component laboratory approval process.
3. Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of PT samples, subject to availability, using each applicable method, in the specified matrix, and provide appropriate documentation to the DoD client.
4. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.
5. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

Project Managers (PMs) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

**Note:** In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work. Unless the work is done directly for the USACE or USDA, this notification is made to the client who is responsible for ensuring notification of the regulating agency.

## **8.2 QUALIFYING AND MONITORING SUBCONTRACTORS**

Whenever a PM becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified network laboratory;
- Firms specified by the client for the task (documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with the company (in JD Edwards): A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation for the requested tests prior to sending samples;
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All intra-company laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. Refer to SOP No. CA-C-S-001, Work Sharing Process.

When the potential subcontract laboratory has not been previously approved, PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

**8.2.1** The QA Manager must ensure that the Subcontracting Approval Form (Figure 8-1)

has been completed and have supporting documentation on file prior to initiation of any work. A letter or e-mail is sent to the lab requesting the following information:

- 8.2.1.1** If a lab is NELAC or A2LA accredited,
  - 8.2.1.1.1** Copy of necessary certifications verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accreditable (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.
  - 8.2.1.1.2** Insurance Certificate. This is required by TestAmerica's Chief Financial Officer.
  - 8.2.1.1.3** USDA soil permit if available.\*\*
- 8.2.1.2** For laboratories accredited by other agencies with an auditing program:
  - 8.2.1.2.1** Copy of necessary certifications verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accreditable (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.
  - 8.2.1.2.2** Insurance Certificate. This is required by TestAmerica's Chief Financial Officer.
  - 8.2.1.2.3** USDA soil permit if available.\*\*
  - 8.2.1.2.4** Description of Ethics and Data Integrity Plan.
  - 8.2.1.2.5** The most recent 2 sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action.
  - 8.2.1.2.6** State Audit with Corrective Action Response.
  - 8.2.1.2.7** Example final report to confirm format is compliant and provides the necessary information. (Minimally, it must be determined that batch QC results are included in the laboratory reports and data is appropriately qualified.)
  - 8.2.1.2.8** A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review (it may need to be sent elsewhere for evaluation). This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously [minimum of 6 months] are grandfathered in.)
  - 8.2.1.2.9** DoD work includes additional requirements as described in Section 8.1 above.
- 8.2.1.3** For laboratories performing tests that are unaccredited or accredited by an agency without an audit program:

- 8.2.1.3.1** A copy of their Quality Assurance Manual (controlled if possible). Ensure data quality limits for relevant methods are acceptable and that training procedures are adequate.
  - 8.2.1.3.2** Copy of necessary certifications (if available) verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accreditable (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.
  - 8.2.1.3.3** Insurance Certificate. This is required by TestAmerica's Chief Financial Officer.
  - 8.2.1.3.4** USDA soil permit if available.\*\*
  - 8.2.1.3.5** Evidence of a current SOP per method. A copy of the first page and signature page of the SOP is acceptable. A table of contents including effective dates may also be acceptable. The SOP can be examined if an on-site audit is performed.
  - 8.2.1.3.6** Description of Ethics and Data Integrity Plan.
  - 8.2.1.3.7** The most recent 2 sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action.
  - 8.2.1.3.8** Example final report to confirm format is compliant and provides the necessary information. (Minimally, it must be determined that batch QC results are included in the laboratory reports and data is appropriately qualified.)
  - 8.2.1.3.9** Statement of Qualification (SOQ) or summary list of Technical Staff and Qualifications – position, education and years of experience.
  - 8.2.1.3.10** DoD work includes additional requirements as described in Section 8.1 above.
  - 8.2.1.3.11** A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review (it may need to be sent elsewhere for evaluation). This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously [minimum of 6 months] are grandfathered in.)
- 8.2.2** Once the information is received by the QA Manager, it is evaluated for acceptability and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site along with the associated documentation and notify the finance group for JD Edwards.

\*\*USDA permit is required if soils less than three feet deep from New York, North Carolina, South Carolina, Georgia, Florida, Tennessee, Alabama, Mississippi, Louisiana, Arkansas, Texas, Oklahoma, New Mexico, Arizona, California, Hawaii, or outside the continental U. S. are

to be analyzed. These samples require special shipping measures; check with the EHS Department. It may be necessary to heat-treat the samples before shipping if the subcontract laboratory does not have a USDA permit; however, some analytes/tests may be irrelevant after heat treatment.

**8.2.3** The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. The company does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

**8.2.4** The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contract Department. Any problems identified will be brought to Corporate QA attention.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints must be posted using the Vendor Performance Report (Form No. CW-F-WI-009).
- Information must be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all network laboratories and Corporate QA and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Lab Directors/Managers, QA Managers and Sales Directors.

### **8.3 OVERSIGHT AND REPORTING**

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. For network laboratories, certifications can be viewed on the company website.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within the network.

The PM will communicate with the subcontracted laboratory to monitor the status of the analyses, facilitate successful execution of the work and ensure the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. The project narrative lists the name, address and methods performed for all subcontract laboratories and network laboratories that provided results included in the final report.

**Note:** The results submitted by a network work sharing laboratory may be transferred electronically and the results reported by the network work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

#### **8.4 CONTINGENCY PLANNING**

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, Corporate QA must be informed, and the QA Manager will be required to verify adequacy of proficiency scores and certifications. The laboratory must also request a copy of the raw data to support the analytical results for the first project submitted to the subcontract laboratory unless the laboratory has NELAC accreditation. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. The QA Manager will request full documentation and qualify the subcontractor under the provisions above. The approval process should be completed within 30 calendar days of subcontracting.



**Figure 8-1.**  
**Example - Subcontracting Laboratory Approval Form (Initial / Renewal)**

**SUBCONTRACTING LABORATORY APPROVAL**

*Reference: Section 8 – Quality Assurance Manual*

Date: \_\_\_\_\_  
Laboratory: \_\_\_\_\_  
Address: \_\_\_\_\_  
Contact and e-mail address: \_\_\_\_\_  
Phone: Direct \_\_\_\_\_ Fax \_\_\_\_\_

Requested Item <sup>3</sup>	Date Received	Reviewed/ Accepted	Date
1. QA Manual <sup>3</sup>			
2. Copy of State Certification <sup>1</sup>			
3. State Audit with Corrective Action Response (or NELAC or A2LA Audit) <sup>3</sup>			
4. Most Recent (and relevant) 2 Sets of WP/WS Reports with Corrective Action Response <sup>1,3</sup>			
5. SOQ or Summary list of Technical Staff and Qualifications <sup>3</sup>			
6. SOPs for Methods to Be Loadshifted <sup>2,3</sup>			
7. USDA Soil Permit			
8. Insurance Certificate			
9. Sample Report <sup>3</sup>			
10. For DoD Work: Statement that Lab quality system complies with QSM.			
11. For DoD Work: Approved by specific DoD Component laboratory approval process.			
11. Description of Ethics Program <sup>3</sup>			

1 - Required when emergency procedures are implemented.

2 - Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable. This requirement may also be fulfilled by supplying a table of SOPs with effective dates.

3 – If the laboratory has NELAC accreditation, Item #s 4 through 10 are not required.

On Site Audit Planned: YES NO If yes, Date Completed: \_\_\_\_\_ By Whom: \_\_\_\_\_

Comments:

Lab Acceptable for Subcontracting Work: YES NO Limitations: \_\_\_\_\_

QA Manager: \_\_\_\_\_ Date: \_\_\_\_\_

☐ Forwarded to Contract Coordinator, by: \_\_\_\_\_ Date: \_\_\_\_\_



## **SECTION 9**

### **PURCHASING SERVICES AND SUPPLIES (NELAC 5.4.6)**

#### **9.1 OVERVIEW**

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Capital expenditures are made in accordance with the Controlled Purchases Procedure, CW-F-S-004. Only one quote is required where the item being purchased is a sole source product. Examples of sole source capital expenditures are laboratory test equipment, client specified purchases and building leases. A minimum of two quotes is required where the opportunity exists to source from more than one vendor. All documentation related to the purchase of capital items will be maintained in the individual CapEx files located in Corporate Purchasing. Data will be held in accordance with the record retention policy.

TestAmerica will enter into formal contracts with vendors when it is advantageous to do so. Contracts will be signed in accordance with the Authorization Matrix Policy, CW-F-P-002. Examples of items that are purchased through vendor contracts are laboratory instruments, consumables, copiers and office supplies. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

Non-capital expenditure items are purchased through the requisition and approval process in JD Edwards or through other TestAmerica authorized methods (approved web-sites, purchasing cards). Labs have the ability to select from the approved vendors in JD Edwards.

#### **9.2 GLASSWARE**

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

#### **9.3 REAGENTS, STANDARDS & SUPPLIES**

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents must meet with the requirements of the specific method and testing procedures for which they are

being purchased. Solvents and acids are pre-tested in accordance with Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

### **9.3.1 Purchasing**

The nature of the analytical laboratory demands that all material used in any of the procedures is of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP.

Many consumable items are maintained in an on-site consignment system, where the items in inventory have been pre-approved for laboratory use. If an item is not included in the on-site consignment system but is needed for laboratory operations, the group leader or designee enters the request into the JD Edwards system. The vendor item catalogue number is entered into the system to specify the required quality. Each request is reviewed and approved by the Laboratory Director or the Technical Director (or designee) prior to purchase.

### **9.3.2 Receiving**

It is the responsibility of the laboratory associate (typically sample receiving personnel) to receive the shipment. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Refer to SOP KNOX-QA-0001 for further details. Material Safety Data Sheets (MSDSs) are kept in the laboratory main hallway and online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

### **9.3.3 Specifications**

There are many different grades of analytical reagents available to the analyst. All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method.

- An expiration date can not be extended if the dry chemical is discolored or appears otherwise physically degraded; the dry chemical must be discarded.
- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

- If the dry chemical is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained electronically in the group share on the local area network with the associated scanned calibration files.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 200 psig or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a conductivity of less than  $1\mu\text{mho/cm}$  (or resistivity of greater than  $1.0\text{ megaohm-cm}$ ) at  $25^{\circ}\text{C}$ . The conductivity is checked and recorded daily. If the water's conductivity is outside the specified limit, the Technical Director must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard. Spiking standards are verified before use and instrument calibration standards are verified with a second source calibration verification standard.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

#### **9.3.4      Storage**

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Table 9-1 details specific storage instructions for reagents and chemicals. Section 22 discusses conditions for standard storage.

### **9.4      PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE**

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Director and/or the Laboratory Director. If they agree with the request the procedures outlined in Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, it is given an instrument name in the LIMS, (e.g. MX), and added to the equipment list described in Section 21 maintained by the QA Department. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (see Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department as specified in the laboratory's procedure for software verification KNOX-IT-0001.. Software certificates supplied by the vendors are filed with IT Department. The manufacturer's operation manual is retained at or near the bench.

## **9.5 SERVICES**

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 21. The need for service is determined by analysts and/or Group Leaders. The service providers that perform the services are approved by the Group Leaders/Technical Director.

## **9.6 SUPPLIERS**

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). The level of control used in the selection process is dependent on the anticipated spend and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report (CW-F-WI-009).

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

#### **9.6.1      New Vendor Procedure**

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (CW-F-WI-007 – refer to Figure 9-2).

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Director are consulted with vendor and product selection that have an impact on quality.

Table 9-1.  
**Storage of Reagents and Chemicals**

Chemical	Storage Requirements
Concentrated Acids and Bases	Stored in the original containers at ambient temperature. All organic acids must be stored separately from inorganic acids. Acids must not be stored with bases.
Bulk Dry Chemicals	Stored in the original containers at ambient temperature. All organic acids must be stored separately from inorganic acids. Acids must not be stored with bases.
Working Solutions Containing Organic Compounds	Stored as per method recommendation/ requirement as defined in the standard operating procedure(s). They are generally stored refrigerated at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .
Working Solutions Containing only Inorganics	Stored at ambient temperature; refrigeration is optional.
Flammable Solvents	Stored in flammable solvent cabinets at ambient temperature.
Non-Flammable Solvents	Stored in solvent cabinets at ambient temperature.

**Figure 9-2**  
**Example – JD Edwards Vendor Add Request Form**



JD Edwards Vendor Add Request Form

Vendor name:	Lab location <u>and</u> individual making request:
Vendor address (remit to):	Vendor phone:
Vendor address (remit to):	Vendor fax:
Contact name:	Product / service provided:

**Reason for Vendor Addition: Check all reasons that apply**

<input type="checkbox"/> Cost Reduction	Estimated Annual Savings \$
<input type="checkbox"/> Replace Current Vendor	Reason?
	Vendor being Replaced?
<input type="checkbox"/> New Product / Service	Describe:
<input type="checkbox"/> ISO Approved ( <u>Required for Aerotech / P&amp;K only</u> )	

**Small Business:**

Does this vendor help us to meet our small business objectives: _____
If yes, which category: _____

**Personal and Ethical Considerations:**

Is there any personal conflict of interest with a TestAmerica employee and the vendor listed above? _____
Have ethical considerations been taken into account in your evaluation of this vendor? _____
_____

**Can this product be sourced from another TestAmerica facility?** \_\_\_\_\_

Please complete form and email to [NCPurchasing@testamericainc.com](mailto:NCPurchasing@testamericainc.com) or fax to (330) 966-9275.

I approve the addition of this vendor:

\_\_\_\_\_  
Purchasing Manager - Patrick Eckman

\_\_\_\_\_  
Corporate Controller - Leslie Bowers

Form No. CW-F-WI-007

## **SECTION 10**

### **SERVICE TO THE CLIENT (NELAC 5.4.7)**

#### **10.1 OVERVIEW**

TestAmerica Knoxville cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements discussed in Section 5. The laboratory has procedures to ensure confidentiality to clients (Section 16 and 26).

**Note:** ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

#### **10.2 SPECIAL SERVICES**

The laboratory's standard procedures for reporting data are described in Section 26. When requested the following special services are provided:

- The laboratory will provide the client or the client's representative reasonable access to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- The laboratory will work with client-specified third party data validators as specified in the client's contract.
- The laboratory will provide the client with all requested information pertaining to the analysis of their samples. An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

#### **10.3 CLIENT COMMUNICATION**

Project managers are an important communication link to the clients. The lab shall inform its clients of any delays in project completion as well as any non-conformances in either sample receipt (refer to Section 24) or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Directors are available to discuss any technical questions or concerns that the client may have.

#### **10.4 REPORTING**

The laboratory will work with the client to produce any special communication reports required by the contract.



## **10.5      CLIENT SURVEYS**

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica's Sales and Marketing teams periodically develop lab and client specific surveys to assess client satisfaction.

## **SECTION 11**

### **COMPLAINTS (NELAC 5.4.8)**

#### **11.1 OVERVIEW**

TestAmerica Knoxville believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that helps to continually improve processes and improve client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, communications, responsiveness, data, reports, invoicing and other functions expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for dealing with both external and internal complaints.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 13 (Corrective Actions) and is documented following the procedure for Nonconformance and Corrective Action (SOP KNOX-QA-0008). Client complaints are recorded as a type of nonconformance memo (NCM) in the Clouseau database, and are summarized in the monthly QA Reports to Management. It is the laboratory's goal to provide a satisfactory resolution to complaints in a timely and professional manner.

#### **11.2 EXTERNAL COMPLAINTS**

An employee that receives a complaint initiates the complaint resolution process and the documentation of the complaint.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving Complaints
- Complaint Investigation and Service Recovery

- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

### **11.3      INTERNAL COMPLAINTS**

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 13. In addition, Corporate management, Sales and Marketing and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 13.

### **11.4      MANAGEMENT REVIEW**

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 17).

## SECTION 12

### CONTROL OF NON-CONFORMING WORK (NELAC 5.4.9)

#### 12.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 13).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is typically discussed with the group supervisor. The supervisor or analyst may elect to discuss it with the Technical Director or have the Project Manager contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's corrective action system described in Section 13. This information is supplied to the client in the form of a case narrative with the report.

Project Managers may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. Such a request would need to be documented as a Nonconformance Memo. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with NELAC (or the analytical method) requirements and the reason.

#### 12.2 RESPONSIBILITIES AND AUTHORITIES

SOP No. CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall, outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of the company's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director, Technical Director, Group Leader or the QA Manager may exceptionally authorize departures from documented procedures or policies depending on the nature and extent of the departure. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures described in Section 13. This information may also

need to be documented on data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility senior laboratory management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, the Customer Service Manager, the Technical Director(s) and the Operations Manager. The Laboratory Director and QA Manager must be included in the notification. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO) and Quality Director within 24 hours.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, COO's – East and West, General Managers and the Quality Directors – East and West have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

### **12.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN**

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

SOP No. CA-L-S-001 distinguishes between situations when it would be appropriate for the laboratory QA Manager and Laboratory Director (or his/her designee) to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting (Section 13) in lieu of the data recall determination form contained in SOP No. CA-L-S-001.

### **12.4 PREVENTION OF NONCONFORMING WORK**

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system (Section 13).

On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

### **12.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)**

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 12.2, Paragraph 5 above.

Prior to suspension/restriction, confidentiality will be respected, and the problem and the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 13 if one has not already been started. A copy of any meeting notes and agreed upon steps must be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (i.e., Project Management, Log-in, etc.). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the nonconformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, QA Manager, Project Manager, Group Leader) shall devise a start-up plan to cover all steps from client notification through compliance and release of reports. The Director of Client Services and Sales and Marketing should be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report as described in Section 13.

## SECTION 13

### CORRECTIVE ACTION (NELAC 5.4.10)

#### 13.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Nonconformance Memos (NCMs) and Corrective Action Reports (CARs) (refer to Figure 13-1).

#### 13.2 DEFINITIONS

- **Correction:** Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions are contained in the method specific SOPs. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.
- **Corrective Action:** The action taken is not only a correction made to the immediate event, but a change in process, procedure or behavior that is required to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

#### 13.3 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigation.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify Systematic Problems before they become serious.
- Identify and track Client complaints and provide resolution (see more on client complaints in Section 11).

**13.3.1 Nonconformance Memo (NCM)** - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP.
- QC outside of limits (non matrix related).

- Isolated Reporting / Calculation Errors.
- Client Complaints.

**13.3.2 Corrective Action Report (CAR)** - is used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Failed or Unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic Reporting / Calculation Errors.

#### **13.4 CLOSED LOOP CORRECTIVE ACTION PROCESS**

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

##### **13.4.1 Cause Analysis**

- Upon discovery of a non-conformance event, the event must be defined and documented. A CAR or NCM must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 13-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Group Leader, Technical Director, or QA Manager (or QA designee) is consulted.

##### **13.4.2 Selection and Implementation of Corrective Actions**

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The CAR or NCM is used for this documentation.

##### **13.4.3 Monitoring of the Corrective Actions**

- The Group Leaders, Operation Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions will be documented and re-evaluated until acceptable resolution is achieved. Group Leaders are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.



- Each NCM is entered into a database for tracking purposes and a monthly summary of all NCMs is printed out for review to aid in ensuring that the appropriate corrective actions have taken effect. CARs are also compiled and reviewed monthly.
- The QA Manager reviews monthly NCMs for trends. Highlights are included in the QA monthly report (refer to Section 17). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

#### **13.4.4      Follow-up Audits**

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements. (Section 16 includes additional information regarding internal audit procedures.)
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

### **13.5      TECHNICAL CORRECTIVE ACTIONS**

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 12 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCM.

Table 13-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to specific method SOPs.

Table 13-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, QAM Sections 20 and 21, and SOP CA-L-S-001 (Internal Investigation of Potential Data Discrepancies and Determination for Data Recall). All corrective actions are reviewed at a minimum monthly by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

### **13.6      BASIC CORRECTIONS**

When mistakes occur in records, each mistake shall be crossed-out, and not erased, deleted, made illegible, or otherwise obliterated (e.g. no white-out), and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

**Figure 13-1.**  
**Example – NonConformance Memo**

## Clouseau Nonconformance Memo



NCM #: <b>08-0006816</b>	
NCM Initiated By: Holly Taj	Classification: <b>Deficiency</b>
Date Opened: 02/22/2007	Status: <b>CLOSED</b>
Date Closed: 03/12/2007	Production Area: GC/MS
	Tests: TO-15
	Lot #'s (Sample #'s): H7 B220202 (12,2,4,5,9),
	QC Batches: ,
Nonconformance: Other (describe in detail)	
Subcategory: See explanation below...	

### Problem Description / Root Cause

<u>Name</u>	<u>Date</u>	<u>Description</u>
Holly Taj	02/22/2007	<p>Upon receipt the following samples were at -1.0 to 0 in Hg. All flow controllers were working within specifications upon receipt.</p> <p>JPV27 can 6589 flow controller K253 pressure -0.9 in Hg.</p> <p>JPV3A can 6359 flow controller K312 pressure +0.3 psig.</p> <p>JPV3E can 1115 flow controller K261 pressure -0.3 in Hg.</p> <p>JPV3K can 2046 flow controller K235 pressure +0.4 psig.</p> <p>JPV3T can 12492, flow controller K227, pressure upon receipt read -1.8 in Hg. the field test data sheet read -28.9 in Hg as the final reading for this sample. The flow controller and guage were working upon receipt to the lab.</p>

### Corrective Action

<u>Name</u>	<u>Date</u>	<u>Corrective Action</u>
Holly Taj	02/22/2007	PM notified. Flow controllers tested.

### Client Notification Summary

<u>Client</u>	<u>Project Manager</u>	<u>Notified</u>	<u>Response</u>	<u>How Notified</u>	<u>Note</u>
ERM Group Inc, The	MCKINNEYJ	02/22/2007	02/22/2007	by telephone	Ted Keffer, 022307 @ 1345
	<u>Response</u>	<u>Response Note</u>			
	Process "as-is"				

### Quality Assurance Verification

<u>Verified By</u>	<u>Due Date</u>	<u>Status</u>	<u>Notes</u>
KHOUNLAVON		Verified/completed	verified group narrative on 3/12/07

**Table 13-1.**

**Example – General Corrective Action Procedures**

<b>QC Activity (Individual Responsible for Initiation/Assessment)</b>	<b>Acceptance Criteria</b>	<b>Recommended Corrective Action</b>
Initial Instrument Blank  (Analyst)	- Instrument response < RL <sup>1</sup> .	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards  (Analyst, Supervisor)	- Correlation coefficient $r \geq 0.990$ for organics and $r \geq 0.995$ for inorganics or - %RSD within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source)  (Analyst, Supervisor)	- % Recovery within acceptance limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards  (Analyst, Data Reviewer)	% Difference (or %Drift) within acceptance limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD)  (Analyst, Data Reviewer)	- % Recovery within limits documented in QuantIMS.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set.
Laboratory Control Sample (LCS) and LCSD (where applicable)  (Analyst, Data Reviewer)	- % Recovery within limits specified in QuantIMS.	- Batch must be re-prepared and re-analyzed. <b>Note:</b> See Method SOPs for further details. For example, if there is insufficient sample or the holding time cannot be met, contact client and report with flags.

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Surrogates  (Analyst, Data Reviewer)	- % Recovery within limits specified in QuantIMS.	- Individual sample must be repeated. The sample batch must be re-extracted/reanalyzed if a QC sample (e.g. method blank) has a surrogate outlier. <b>Note:</b> See Method SOPs for further details. For example, if there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Method Blank (MB)  (Analyst, Data Reviewer)	< Reporting Limit <sup>1</sup>	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.

**Note:**

1. See specific method SOPs for acceptance criteria. Concentrations up to five times the reporting limit may be allowed for the common laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates. Some program requirements (e.g. DOD QSM, AFCEE, and Ohio VAP) have more stringent requirements that must be followed when performing work for those programs. For example, DOD QSM and AFCEE require the Method Blanks < ½ Reporting Limit with common lab contaminants < Reporting Limit. Ohio EPA VAP requires all analytes of concern to be less than the RL.

## SECTION 14.0

### PREVENTIVE ACTION (NELAC 5.4.11)

#### 14.1 OVERVIEW

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes TestAmerica Knoxville's commitment to its Quality Assurance (QA) program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc.

The monthly Quality Assurance Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's Corrective Action process (Section 13) is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

**14.1.1** The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process, management review, and through the review and revision of the Quality Assurance Manual and/or laboratory SOPs.

**14.1.2** Any Preventive Actions undertaken or attempted shall be taken into account during the Annual Management Review (Section 17). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program will provide management a measure for evaluation.

## **14.2 MANAGEMENT OF CHANGE**

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. This is routinely managed by the Laboratory Director with input from the General Manager, the QA Manager, Operations Manager, Technical Directors, Group Supervisors, and IT support staff. In this process, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. When required by the Laboratory Director, this process is documented according to SOP CA-Q-S-003, Management of Change.

## SECTION 15.0

### CONTROL OF RECORDS (NELAC 5.4.12)

TestAmerica Knoxville maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Refer to SOP KNOX-AD-0001, Record Retention and Document Storage for further details.

#### 15.1 OVERVIEW

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 15-1. Quality records are maintained by the Quality Assurance (QA) Manager on a designated share on the local area network, which is backed up as part of the regular network backup. Hardcopy quality records are stored in fireproof filing cabinets. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by Group Supervisors, Project Assistants and the local IT staff.

**Table 15-1. Record Index<sup>1</sup>**

Technical Records	Official Documents	QA Records	Project Records	Administrative Records
<b>Retention:</b> 5 Years from analytical report issue*	5 Years from document retirement date*	5 Years from archival* <b>Data Investigation:</b> 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)	5 Years from analytical report issue*	<b>Personnel:</b> 7 Years (HR Records must be maintained as per Policy CW-L-P-001) <b>Finance:</b> See Accounting and Control Procedures Manual
Raw Data	Quality Assurance Manual (QAM)	Internal and External Audits/ Responses	Sample receipt and COC Documentation	Finance and Accounting
Logbooks <sup>2</sup>				
Standard Certificates	QA and Technical Policies	Certifications	Contracts and Amendments	EH&S Manual, Permits, Disposal Records
Analytical Records	SOPs	Corrective/Preventive Action	Correspondence	Employee Handbook
Lab Reports		Management Reviews	QAPP	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)
		Method & Software Validation, Verification data	SAP	
		Data Investigation	Telephone Logbooks	Administrative Policies



Technical Records	Official Documents	QA Records	Project Records	Administrative Records
			Lab Reports	Technical Training Records

<sup>1</sup> Record Types encompass hardcopy and electronic records.

<sup>2</sup> Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).

\* Exceptions listed in Table 15-2.

All records are legible and stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or the Iron Mountain data storage facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 15-2 have lengthier retention requirements and are subject to the requirements in Section 15.1.3. Policy CW-L-P-001 (Record Retention) provides additional information on record retention requirements.

#### 15.1.1 **Programs with Longer Retention Requirements**

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 15-3 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data. Refer to SOP KNOX-AD-0001, Document Storage and Record Retention for details on how records are retained for these programs.

**Table 15-2. Special Record Retention Requirements**

<b>Program</b>	<b><sup>1</sup>Retention Requirement</b>
Drinking Water – All States	10 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
NY Potable Water NYCRR Part 55-2	10 years
Ohio EPA VAP	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

<sup>1</sup>Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

**15.1.2** All records are held secure and in confidence. Records maintained at the laboratory are located in the scanning room and the reporting room as hard copy and on a designated local area network server as electronic copy as described in KNOX-AD-0001, Document Storage and Record Retention. In and out cards are used to document temporary removal of hardcopy lot folders from shelves.

**15.1.3** The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, see section 20.12.1 'Computer and Electronic Data Related Requirements' for more information.

**15.1.4** The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. A copy of the chain of custody is stored with the invoice, associated data review checklists, and other lot specific documents as described in SOP KNOX-AD-0001. The chain of custody would indicate the name of the sampler.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set). Instrument data is stored electronically with the Lot raw data for samples and the associated quality control samples. Instrument calibration data is stored electronically by analytical group, method, instrument and date. Run logs are maintained for each instrument; a copy of each day's run log or instrument sequence is stored to aid in reconstructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks.
- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for scanning the records is discussed in SOP KNOX-AD-0004, Data Reporting, and KNOX-AD-0001, Document Storage and Record Retention.
- Also refer to Section 20.13.1 'Computer and Electronic Data Related Requirements'.

## **15.2 TECHNICAL AND ANALYTICAL RECORDS**

**15.2.1** The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement (refer to Section 15.1). The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for performance of each analysis and checking of results.

**15.2.2** Observations, data and calculations are recorded at the time they are made and are identifiable to the specific task.

**15.2.3** Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include (previous discussions relate

where most of this information is maintained – specifics may be added below):

- laboratory sample ID code;
- Date of analysis and time of analysis is required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. The date and time may also be recorded in the associated logbook or benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, lab sample ID codes, volumes, weights, instrument printouts, calculations, and reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- method performance criteria including expected quality control requirements. These are indicated in the LIMS or in the associated analytical SOPs

### **15.3      LABORATORY SUPPORT ACTIVITIES**

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures.

#### **15.3.1      Sample Handling Records**

Sample handling and tracking is discussed in Section 24. Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

### **15.4      ADMINISTRATIVE RECORDS**

The laboratory also maintains the administrative records in either electronic or hard copy form. See Table 15-1.

### **15.5      RECORDS MANAGEMENT, STORAGE AND DISPOSAL**

**15.5.1**      All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available to the accrediting body upon request.

**15.5.2** All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

**15.5.3** Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

**15.5.4** TestAmerica Knoxville has a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and project records. Laboratory notebooks are sequentially numbered by QA and issued to the analytical group as needed. Bench sheets are filed and archived with the project raw data. Completed logbooks are returned to QA for archive storage.

**15.5.5** Records are considered archived when moved to the archive server on the local area network or when logbooks are turned in to QA for storage. Access to archived hard-copy information is documented with in/out cards. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to the data is limited to laboratory and company employees.

**15.5.6** In the event that the laboratory transfers ownership or goes out of business, TestAmerica Knoxville shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

**15.5.7 Records Disposal**

**15.5.7.1** Records are removed from the archive and disposed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration.

**15.5.7.2** Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

**15.5.7.3** If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required. [Refer to Policy No. CW-L-P-001 (Records Retention).]

## SECTION 16

### AUDITS (NELAC 5.4.13)

#### 16.1 OVERVIEW

Audits measure laboratory performance and insure compliance with accreditation/certification and project requirements. Audits specifically provide management with an on-going assessment of the quality of results produced by the laboratory, including how well the policies and procedures of the QA system and the Ethics and Data Integrity Program are being executed. They are also instrumental in identifying areas where improvement in the QA system will increase the reliability of data. There are two principle types of audits: Internal and External. Internal audits are performed by laboratory or corporate personnel. External audits are conducted by regulators, clients or third-party auditing firms. In either case, the assessment to program requirements is the focus.

**Table 16-1. Audit Types and Frequency**

Internal Audits	Description	Performed by	Frequency
	Analyst & Method Compliance	QA Department or Designee	- 100% of all methods over a two year period. - 100% of all analysts annually.
	Instrument	QA Department or Designee	100% of all organic instruments and any inorganic chromatography instruments. - Every two years.
	Work Order/ Final Report	QA Department or Designee	- 1 complete report each month.
	Support Systems	QA Department or Designee	- Annual for entire labs support departments & equipment (e.g., thermometers, balances), can be divided into sub-sections over the course of the year.
	Performance Audits (Double-Blind PTs)	Corporate QA, Laboratory QA Department or Designee	- As needed.
	Special	QA Department or Designee	- As Needed
External Audits	Description	Performed by	Frequency
	Program / Method Compliance	Regulatory Agencies, Clients, accreditation organizations	- As required by program and/or clients needs
	Performance Audits	Provided by a third party.	- As required by a client or regulatory agency. Generally provided semi-annually through the analysis of PT samples.

#### 16.2 INTERNAL AUDITS

Annually, the laboratory prepares a schedule of internal audits to be performed throughout the year. As previously stated, these audits verify and monitor that operations continue to comply with the requirements of the laboratory's QA Manual and the Corporate Ethics Program, and the



DoD QSM. A schedule of the internal audits is maintained by the QA Manager in the *Internal Audit Workbook*. An example can be found in Attachment 1.

It is the responsibility of the QA Manager to plan and organize audits in consideration of the laboratory work load and the department personnel schedules so that all pertinent personnel and operations are thoroughly reviewed. When designees (other than QA department personnel & approved by the QA Manager), perform audits, the QA Manager shall insure that these persons do not audit their own activities except when it can be demonstrated that an effective audit will be carried out. In general, the auditor:

- is neither the person responsible for the process being audited nor the immediate supervisor of the person responsible for the project/process.
- Is free of any conflicts of interest.
- Is free from bias and influences that could affect objectivity.

Laboratory personnel (e.g., supervisors and analysts) may assist with both method and support system audits as long as the items listed in the above paragraph are observed. These audits are conducted according to defined criteria listed in the checklists of the *Internal Audit Workbook*. These personnel must be approved by the QA Manager and must complete the audit checklists in their entirety. This process introduces analyst experience and insight into the laboratory's auditing program.

The auditor must review the previous audit report and identify all items for verification of corrective actions. A primary focus will be dedicated to the ability of the laboratory to correct root-cause deficiencies and that the corrective action has been implemented and sustained as documented.

### **16.2.1 Systems**

An annual systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems, client and State requirements. This audit is performed in portions throughout the year through method, analyst, instrument, work order/final report and support system audits. Audits are documented and reported to management within 1 week of their performance. Systems audits cover all departments of the facility, both operational and support. The multiple audits are compiled into one systems audit package at the end of the year (*Internal Audit Workbook*).

#### **16.2.1.1 Method, Analyst, Instrument and Work Order/Final Report Audits**

Procedures for the method compliance, analyst, instrument and work order/final report audits are incorporated by reference to SOP No. CA-Q-S-004, Method Compliance and Data Authenticity Audits. These audits are not mutually exclusive. For example, the performance of a method audit will also cover multiple analysts and instruments. The laboratory's goal is to annually review all analysts and review all instruments every two years as described in SOP No. CA-Q-S-004. The laboratory will also audit all methods within a two year time period and audit a minimum of one Work Order/Final Report from receiving through reporting on a monthly basis.



#### **16.2.1.2 Support Systems**

Support system audits are performed to ensure that all departments & ancillary equipment are operating according to prescribed criteria. Support system audits include the review of both non-analytical and operational departments. Support equipment audits (e.g., metrology items) include the review of balance calibrations, weight calibrations water quality testing, etc.. Non-analytical may include sample receiving and bottle preparation. These types of support audits ensure that the operations are being performed to support ethical data as well as ensuring the accuracy and precision of the utilized equipment.

These audits can be performed in portions throughout the year or in one scheduled session. However, the audit schedule must document that these aspects are reviewed annually. Many of the metrology systems are considered to be surveillance activities that can be monitored by QA personnel or delegated to specified department personnel. These surveillance activities are performed on a semi-annual basis unless issues warrant a greater frequency or previous audits continually showing no deficiencies allow the frequency to be reduced to once a year.

An example audit checklist can be found in Attachment 2. Instructions for reporting findings are included in the *Internal Audit Workbook*. In general, findings are reported to management within 1 week of the audit and a response is due from management within 30 days.

#### **16.2.2 Performance Audits**

Corporate QA may arrange for double blind PT studies to be performed in the laboratories. Results are given to Management and Corrective actions of any findings are coordinated at each facility by the QA Managers and Laboratory Directors. These studies are performed on an as needed basis. They may be performed when concerns are raised regarding the performance of a particular method in specific laboratories, periodically to evaluate methods that may not normally be covered in the external PT program or may be used in the process of developing best practices. The local QA Manager may also arrange for PT studies on an as needed basis. (Refer to Section 16.3.2 for additional information on Performance Audits.)

#### **16.2.3 Special Audits**

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

### **16.3 EXTERNAL AUDITS**

TestAmerica facilities are routinely audited by clients and external regulatory authorities. External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. The laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. This time frame is generally 30 days.

TestAmerica Knoxville cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

#### **16.3.1 Confidential Business Information (CBI) Considerations**

During on-site audits, on-site auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

#### **16.3.2 Performance Audits**

The laboratory is involved in performance audits conducted semi-annually through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Water Supply, Water Pollution, Soil, Ambient Air, and Mixed Analyte (DOE).

- It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Further, where PT samples present special or unique problems in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.
- PTs generally do not have holding times associated with them. In the absence of any holding time requirement, it is recommended that the holding time begin when the PT sample is prepared according to the manufacturers instructions. Holding times apply to full volume PT samples only if the provider gives a meaningful "sampling date". If this is not provided, it is recommended that the date/time of opening of the full volume sample be considered the beginning of holding time.
- Login will obtain the COC information from the documentation provided with the PTs with review by QA or other designated staff.
- Vials will be prepared as required in the instruction set provided with the samples. After preparation to full volume the sample must be spiked, digested, concentrated, etc., as would be done for any normal sample requiring similar analysis.

- PT samples will not undergo multiple preps, multiple runs, multiple methods (unless being used to evaluate multiple methods), multiple dilutions, UNLESS this is what would be done to a normal client sample (e.g., if a client requests, as PT clients do, that we split VOA coeluters, then dual analysis IS normal practice).
- The type, composition, concentration and frequency of quality control samples analyzed with the PT samples shall be the same as with routine environmental samples.
- Instructions may be included in the laboratory's SOPs for how low level samples are analyzed, including concentration of the sample or adjustment of the normality of titrant. When a PT sample falls below the range of the routine analytical method, the low-level procedure may be used.
- No special reviews shall be performed by operation and QA, UNLESS this is what would be done to a normal client sample. To the degree that special report forms or login procedures are required by the PT supplier, it is reasonable that the laboratory WOULD apply special review procedures, as would be done for any client requesting unusual reporting or login processes.
- Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

#### **16.4      AUDIT FINDINGS**

Internal Audits are documented in summary report(s) providing the findings for each laboratory area and recommended corrective actions to the appropriate Group Leader. External Audits are typically documented in a similar fashion based on findings provided in the external audit report. The laboratory corrective action is compiled by QA and the response to the external audit is signed by the QA Manager and the Laboratory Director. The laboratory is expected to prepare a response to audit findings within 30 days of receipt of an audit report unless the report specifies a different time frame. The response may include action plans that could not be completed within the 30 day timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Responsibility for developing and implementing corrective actions to findings is the responsibility of the Group Leader where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

The procedures must be in accordance to SOP No. CA-L-S-001, Internal Investigations of Data Discrepancies and Determination of Data Recall.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

Figure 16-1.

Example - Internal Audit Workbook



TestAmerica Knoxville  
Last Updated: 9/10/2007

Workbook Instruction No. CA-Q-WI-011

Internal Audit Workbook  
Summary Page

**Note:** Click on the [\(Summary Page\)](#) to located on each audit sheet to return to this page.

\* The lab may choose to audit these areas with each method/analyst/instrument audit. The auditor must document on the checklist that this item is audited as part of the <defined> audit.

Area Audited	Audit Type	Audit Cycle	Scheduled	Date Audited	Date Closed	Comments
(Click on the Area to get to that Spreadsheet)						
Balances	System	6 mo				
Temperature Logs/Thermometers	System	6 mo				
Sample Storage and Disposal	System	1 yr				
Maintenance Logs *	System	6 mo				
Volatile Storage Blanks	System	6 mo				
Lab Water Quality Testing	System	6 mo				
Sample Log In	System	1 yr				
Shipping Procedures	System	1 yr				
SOP & Document Distribution System	System	1 yr				
Statistical Process Control	System	1 yr				
Data Review System	System	1 yr				
Final Report Generation	System	1 yr				
Standards/Reagents *	System	6 mo				
Manual Integration *	System	1 yr				
Corrective Action System	System	1 yr				
Training Records	System	6 mo				
MDLs	System	1 yr				
SOPs - Prep/Review/Update Process	System	1 yr				
Purchasing/Procurement	System	1 yr				
Eppendorf/Diluter/Dispenser Calibration Check	System	6 mo				
Subcontract Lab Approval	System	1 yr				
Customer Complaint System	System	1 yr				
Methods	Method	2 yr				

Figure 16-2.

Example – Internal Audit System Checklist: Corrective Actions



(Summary Page)

TestAmerica <Location>

INTERNAL AUDIT - Corrective Actions

[ Printed Name(s) or Date(s) ]

Area Audited: \_\_\_\_\_  
Auditor: \_\_\_\_\_  
Date: \_\_\_\_\_  
Persons Contacted During Audit: \_\_\_\_\_  
Date Reported to Department Manager: \_\_\_\_\_  
Reported To: \_\_\_\_\_  
Date Reported to Lab Director/Manager: \_\_\_\_\_  
Reported To: \_\_\_\_\_  
Date Response Due: \_\_\_\_\_  
Response Received and Accepted by QA Manager: \_\_\_\_\_  
Associated Corrective Action Report Number(s): \_\_\_\_\_  
Scheduled Follow-up: \_\_\_\_\_

Item	Requirement	Ref.	Y	N	NA	Evidence/Comments	Follow Up
1	Does the laboratory have a corrective action program in place?	5.4.10.1					
2	Does the laboratory have a current corrective action SOP or is this information in the QA Manual?	5.4.10.1					
3	Do all laboratory personnel have documented training and access to initiate corrective actions?	5.4.10.1					
4	Are causes clearly identified by department, staff name, scope of issue (how many reports affected)?	5.4.10.6					
5	Is a root cause for the issue identified?	5.4.10.2					
6	Is a corrective action (plan) clearly described?						
7	Was the corrective action fully implemented?						
8	Is documentation (if applicable) completed as specified by the corrective action (training, revised SOP, etc)						
9	Has a follow-up assessment been conducted to verify the corrective action was successful?						
10	Are corrective actions reviewed on a regular basis by management?	5.4.10.6a 5					
11	Is there a defined distribution flow for corrective action notification, review, closure, and follow-up?	5.4.10.6a					
12	Are non-conformances reviewed on a regular basis and used, if necessary, to initiate root cause corrective actions?						
13	Does the lab have a documented procedure for QC corrective action (i.e., documented within each method / parameter SOP or in the QA Manual)?	4.10.1					
14	<b>Verify Corrective Actions from previous systems audits. List Items:</b>						
15							
16							
17							

Auditor Signature: \_\_\_\_\_

Primary Reference(s): Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices  
NELAC Standard, June 2003  
DoD Quality Systems Manual, Version 3, January 2006  
EPA Manual for the Certification of Laboratories Analyzing Drinking Water

## SECTION 17

### MANAGEMENT REVIEWS (NELAC 5.4.14)

#### 17.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director for review and comments. The final report shall be submitted to the Technical Directors, Operation Manager, Group Leaders, Laboratory Director, as well as the appropriate Quality Director and General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. At a minimum, the report content will contain the items listed below. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

The TestAmerica QA Report template is comprised of a discussion of three key QA issues facing the laboratory and ten specific sections (Figure 17-1):

- **Metrics:** Describe actions or improvement activities underway to address any outlying quality metrics that have been reported in the monthly Quality System Metrics Table.
- **SOPs:** Report SOPs that have been finalized and report status of any outstanding SOP reviews.
- **Corrective Actions:** Describe highlights and the most frequent cause for report revisions and corrective/preventive action measures underway. Include a discussion of any recalls handled at the lab level as per Section 6.2.2 in the Investigation/Recall SOP (SOP: CA-L-S-001). Include a section for client feedback and complaints. Include both positive and negative feedback. Describe the most serious client complaints and resolutions in progress.
- **MDLs and Control Limits:** Report which MDLs/ MDL verifications are due. Report the same for Control Limits.
- **Audits:** Report Internal and External Audits that were conducted. Include all relevant information such as which methods, by whom, corrective actions needed by when and discuss unresolved audit findings.
- **Performance Testing (PT) Samples:** Report the PT tests that are currently being tested with their due dates, report recent PT results by study, acceptable, total reported and the month and year.
- **Certifications:** Report on any certification programs being worked on by due date, packages completed. Describe any issues, lapses, or potential revocations.
- **Regulatory Updates:** Include information on new state or federal regulations that may impact the laboratory. Report new methods that require new instrumentation, deletion of methods, changes in sampling requirements and frequencies etc.
- **Miscellaneous:** Include any issues that may impact quality within the laboratory.
- **Next Month:** Report on plans for the upcoming month.



- **Lab Director Comments Section:** This section gives the Laboratory Director the opportunity to comment on issues discussed in the report and to document plans to resolve these issues. Unresolved issues that reappear in subsequent monthly reports must be commented on by the Laboratory Director.
- **Quality Systems Metrics Table:** The report also includes statistical results that are used to assess the effectiveness of the quality system. Effective quality systems are the responsibility of the entire laboratory staff. Each laboratory provides their results in a template provided by Corporate QA (Figure 17-2).

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The VP-QA/EHS prepares a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Analytical Division Senior Management Team and General Managers.

## **17.2      ANNUAL MANAGEMENT REVIEW**

The senior lab management team (Laboratory Director, Technical Directors, QA Manager, and the Customer Service Manager) conducts an annual review of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This review uses information generated during the preceding year to assess the “big picture” by ensuring that routine quality actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review (refer to Section 17.1) should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior Senior Management team meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources.
  - Adequacy of policies and procedures.
  - Future plans for resources and testing capability and capacity.



- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

The annual review includes the previous 12 months. Based on the annual review, a report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

The QA Manual is also reviewed at this time and revised to reflect any significant changes made to the quality systems.

### **17.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS**

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The Corporate Data Investigation/ Recall SOP shall be followed (SOP No. CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

The Chairman/CEO, President/CEO, COOs and Quality Directors receive a monthly report from the VP of Quality and EHS summarizing any current data integrity or data recall investigations as described in SOP No. CA-L-S-001. The General Manager's are also made aware of progress on these issues for their specific labs.

**Figure 17-1.**

**Example - QA Monthly Report to Management**

LABORATORY: x  
PERIOD COVERED: Month/Year  
PREPARED BY: x      DATE: Month Day, Year  
DISTRIBUTED TO: xx (Include LD, GM, QA Director, etc...)

**THREE KEY ISSUES FOR MONTH:**

*Include a discussion of three key issues that were focused in on this month.*

1. x
  2. x
  3. x
- 

**1. METRICS**

*Describe actions or improvement activities underway to address any outlying quality metrics.*

**2. SOPs**

See Tab for SOP specifics.

The following SOPs were finalized (or reviewed for accuracy): (See Tab)

The following SOPs are due to QA: xx

In QA to complete: xx

**3. CORRECTIVE ACTION**

Highlights: xx

Revised Reports:

*Describe the most frequent cause for report revisions and corrective/preventive action measures underway.*

Data Investigations/Recalls (Corporate Data Investigation/Recall SOP ) :

*Include a discussion of any recalls handled at the lab level as Corp SOP.*

Client Feedback and Complaints:

*Include both positive and negative feedback.*

Describe the most serious client complaints) and resolutions in progress.

**4. MDLs AND CONTROL LIMITS**

MDLs Due:

Control Limits Due:

## 5. AUDITS

### INTERNAL AUDITS

*Discuss Any Outstanding Issues (or Attach Summary):*

### EXTERNAL AUDITS

*Discuss Any Outstanding Issues (or Attach Summary):*

## 6. PT SAMPLES

The following PT samples are now in house (Due Dates):

xx

## 7. CERTIFICATIONS

Certification Packages Being Worked On (Include Due Date):

x

Describe any issues, lapses, or potential revocations.

## 8. REGULATORY UPDATE

*Include information on new state or federal regulations that may impact the laboratory – new methods that require new instrumentation, deletion of methods, changes in sampling requirements or frequencies, ...*

## 9. MISCELLANEOUS

*Include any issues that may impact quality within the laboratory.*

## 10. NEXT MONTH

*Items planned for next month.*

LAB DIRECTOR COMMENTS AND PLANNED CORRECTIVE ACTIONS:

LAB DIRECTOR REVIEW:

DATE:

**Figure 17-2.**

**Example - Laboratory Metrics Categories**

# Reports for month
# Reports revised due to lab error
<b>% Revised Reports</b>
<b># of Data Recall Investigations</b>
<b># of Reports Actually Recalled</b>
# Corrective Action Reports
# Corrective Action Reports still open
<b>Total Number of Unresolved Open Corrective Action Reports</b>
<b>% of Unresolved Open Corrective Action Reports</b>
# Reports independent QA reviewed
<b>% QA Data Review: Reports</b>
# Technical staff (Analysts/technicians, including Temps)
# of Analyst work product reviewed year-to-date
# of Analytical instruments w/electronic data file storage capability
# of Analytical instruments reviewed for data authenticity year-to-date
<b>% Analyst/Instrument Data Authenticity Audits</b>
<b># Client Complaints</b>
<b># Client Compliments</b>
# of planned internal audits
# of planned internal method audits performed year-to-date
<b>% Annual Internal Audits Complete</b>
<b># of Open Internal Audit Findings Past Due</b>
<b>Total Number of External Audit Findings</b>
<b># of Open External Audit Findings Past Due</b>
<b>% External Audit Findings Past Due</b>
# of PT analytes participated and received scores
# of PT analytes not acceptable
<b>% PT Cumulative Score</b>
<b># PT Repeat Analyte Failures Cumulative (analyte failed more than once in 4 consecutive studies by PT Type) (only applies to failed analytes)</b>
# SOPs

# SOPs Reviewed/revised within 24 months
# Methods or Administrative procedures without approved SOPs
<b>SOP Status</b>
<b>Method certification Losses due to performance/audit issues</b>
<b>Hold Time Violations due to lab error</b>
<b>Date of Last Comprehensive Ethics Training Session</b>
<b># Staff that haven't Received Comprehensive Ethics Training (&gt;30 Days From Employment Date)</b>
<b>MDL Status (Good, Fair, or Poor) &gt;90%, &gt;70%, &lt;70%</b>
<b>Training Documentation Records (Good, Fair, or Poor)</b>
<b>LQM Revision/review Date</b>
<b>QAM Updated to New Integrated Template</b>
<b>Last Annual Internal Audit Date (Opened, Closed)</b>
<b>Last Management QS Review Date</b>
#SOPs required for 12 month review cycle (DOD or drinking water)
#SOPs for 12 month cycle/revised within 12 months (Includes QS and Methods Listed in QSM)
<b>12 month % SOP Status</b> (Includes QS and Methods Listed in QSM)

## **SECTION 18**

### **PERSONNEL (NELAC 5.5.2)**

#### **18.1      OVERVIEW**

TestAmerica's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Appendix 2.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

#### **18.2      EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL**

TestAmerica makes every effort to hire analytical staff that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions may be made based upon the technical nature of the position and the individual's experience and abilities. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (also see Section 4 for

position descriptions/responsibilities) or are available from the laboratory Human Resources representative.

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience or 5 years of prior analytical experience
Technical Directors/Department Managers – <b>General</b>	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry  An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Group Leader, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

### 18.3 **TRAINING**

TestAmerica is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Refer to EH&S Manual	All

Required Training	Time Frame	Employee Type
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	30 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to “Demonstration of Capability” in Section 20.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in the Laboratory Training SOP KNOX-QA-0009.

#### **18.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM**

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established an Ethics Policy No. CA-L-P-001 and an Ethics Statement/Agreement (Appendix 1). All initial and annual training is documented by signature on the signed Ethics Policy and Code of Ethical Conduct



demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy (Appendix 1).
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g., peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion).
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

## **SECTION 19**

### **ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS (NELAC 5.5.3)**

#### **19.1 OVERVIEW**

TestAmerica Knoxville is a 28,000 ft<sup>2</sup> secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, specialty organics sample preparation, specialty organics analysis, metals sample preparation, metals analysis, inorganic sample analysis, and administrative functions.

#### **19.2 ENVIRONMENT**

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may effect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity controls, temperature controls, and voltage conditioning with an uninterruptible backup power supply.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels (refer to Section 12).

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

### **19.3      WORK AREAS**

There is effective separation between neighboring areas when the activities therein are incompatible with each other. For example:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

### **19.4      FLOOR PLAN**

A floor plan can be found in Appendix 3.

## **19.5      BUILDING SECURITY**

Building access codes and alarm codes are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of TestAmerica Knoxville. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

## SECTION 20.0

### TEST METHODS AND METHOD VALIDATION (NELAC 5.5.4)

#### 20.1 OVERVIEW

TestAmerica Knoxville uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

#### 20.2 STANDARD OPERATING PROCEDURES (SOPs)

TestAmerica Knoxville maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory (refer to Section 6 on Document Control):

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for preparation, review, revision and control are incorporated by reference to SOPs: **CW-Q-S-002** "Writing a Standard Operating Procedure" SOPs KNOX-QA-0018 "Preparation and Management of SOPs," and KNOX-QA-0011 "Document Control and Distribution."
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

#### 20.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP. Refer to the corporate SOP CW-Q-S-002 "Writing a Standard Operating Procedure" for content and requirements of technical and non-technical SOPs. Also refer to SOP KNOX-QA-0018 "Preparation and Management of SOPs."

**Note:** If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from

the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

## **20.4      SELECTION OF METHODS**

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology. The methods selected must be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

### **20.4.1      Sources of Methods**

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

In general, TestAmerica Knoxville follows procedures from the referenced methods shown below in 20.4.1.1.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

**20.4.1.1** The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.
- Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.
- Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.

- Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup>/19<sup>th</sup>/20<sup>th</sup> edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.
- Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.
- Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)
- Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

#### **20.4.2 Demonstration of Capability**

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

- 20.4.2.1** A demonstration of capability is performed whenever there is a change in instrument type, method or personnel.
- 20.4.2.2** The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures (refer to Section 15, Control of Records).
- 20.4.2.3** The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (e.g., retention time window study).

**Note:** In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method).
- The reporting limit is set at or above the first standard of the curve for the analyte.
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: (e.g., *Reporting Limit based on the low standard of the calibration curve.*)
- Refer to Section 12 (Control of Non-Conforming Work).

#### **20.4.3 Initial Demonstration of Capability (IDOC) Procedures**

- 20.4.3.1** A quality control sample shall be obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.
- 20.4.3.2** The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by the method or the laboratory SOP.
- 20.4.3.3** At least four aliquots shall be prepared (including any applicable routine clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).
- 20.4.3.4** Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviation for each parameter of interest.
- 20.4.3.5** When it is not possible to determine the mean and standard deviation, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the laboratory SOP.
- 20.4.3.6** Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.
- 20.4.3.7** When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:



- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 20.4.3.3 above.
- Beginning with 20.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 20.4.3.1 above.

**20.4.3.8** Refer to SOP KNOX-QA-0009, Personnel Orientation and Training for further details.

A certification statement (see Figure 20-1 for an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training file.

## **20.5      LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS**

Any new method developed by the laboratory must be fully defined in an SOP/Methods Manual (Section 20.2) and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method. The information included in the checklist below (Figure 20-2) is needed before samples are accepted for analysis by a new method.

## **20.6      VALIDATION OF METHODS**

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled. (From 2003 NELAC Standard)

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

### **20.6.1      Method Validation and Verification Activities for All New Methods**

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

#### **20.6.1.1      Determination of Method Selectivity**

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

#### **20.6.1.2      Determination of Method Sensitivity**

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. The laboratory determinations of MDLs are described in Section 20.7.

#### **20.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)**

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

#### **20.6.1.4 Determination of Interferences**

A determination that the method is free from interferences in a blank matrix is performed.

#### **20.6.1.5 Determination of Range**

Where appropriate, a determination of the applicable range of the method must be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

#### **20.6.1.6 Determination of Accuracy and Precision**

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

#### **20.6.1.7 Documentation of Method**

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

#### **20.6.1.8 Continued Demonstration of Method Performance**

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

## **20.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)**

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 20.7.10). The analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. The replicates may be prepared and analyzed over a period of time (e.g. over multiple batches) or they may be performed within the same batch. If more than seven replicates are used, care must be taken to ensure the appropriate t-value multiplier is applied.

**20.7.1** MDL's are initially performed for each individual instrument and non-microbiological method analysis. Unless there are requirements to the contrary, the laboratory will use the highest calculated MDL for all instruments used for a given method as the MDL for reporting purposes. This MDL is not required for methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report values to the MDL. For titration and gravimetric methods where there is no additional preparation involved, the MDL is based on the lowest discernable unit of measure that can be observed.

**20.7.2** MDL's must be run against acceptable instrument QC, including ICV's and Tunes. This is to insure that the instrument is in proper working condition and falsely high or low MDL's are not calculated.

**20.7.3** Use only clean matrix which is free of target analytes (e.g., laboratory reagent water, Ottawa Sand) unless a project specific MDL is required in a field sample matrix.

**20.7.4** The Reporting Limit (also referred to as Limit of Quantitation or LOQ) should generally be between 2 and 5 times the MDL. If the MDL is being performed during method development, use this guideline to determine the Reporting Limit for the analysis. The Limit of Quantitation for samples from Wisconsin is 3.333 times the limit of detection. For work done in support of the DoD Quality Systems Manual, the RL must be  $\geq 3 \times$  MDL. If a sample is diluted, the reported MDL is adjusted according to the dilution factor.

**20.7.5** The calculated MDL cannot be not greater than the spike amount.

**20.7.6** If the most recent calculated MDL does not permit qualitative identification of the analyte then the laboratory may use technical judgment for establishing the MDL (e.g., calculate what level would give a qualitative ID, compare with IDL (20.8), spike at a level where qualitative

ID is determined and assign that value as MDL, minimum sensitivity requirements, standard deviation of method blanks over time, etc.). Refer to SOP KX-QA-001 Method Detection Limits for further details.

Each of the 7 spikes must be qualitatively identifiable (e.g., appear in both columns for dual column methods, characteristic ions for GCMS mass spectra, etc). Manual integrations to force the baseline for detection are not allowed unless all client sample data is manually reviewed in detail to confirm target analytes are present or not. Note that this is not an allowance for inappropriate manual integration. These integrations need to be documented and reviewed as any manual integration. MDL studies for work in support of Ohio EPA VAP must be spiked at or below the reporting limit.

**20.7.7** The initial MDL is calculated as follows:

$$\text{MDL} = t_{(n-1, 1-\alpha = 0.99)} \times (\text{Standard Deviation of replicates})$$

where  $t_{(n-1, 1-\alpha = 0.99)} = 3.143$  for seven replicates.

**20.7.8** Subsequent to the initial MDL determination, periodic MDL verification, confirmation or determinations may be performed by the procedure in 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices (e.g., method blanks over time, single standard spikes that have been subjected to applicable sample prep processes, etc.). Refer to SOP KX-QA-001 "Method Detection Limits" for further details.

**20.7.9** Because of the inherent variability in results outside of the calibration range, TestAmerica does not recommend the reporting of results below the lowest calibration point in a curve; however, it is recognized that some projects and agencies require the reporting of results below the RL. Any result that falls between the MDL and the Reporting Limit, when reported, will be qualified as an estimated value.

**20.7.10** Detections reported down to the MDL must be qualitatively identified.

**20.7.11** MDLs and Reporting Limits are adjusted in LIMS based on moisture content and sample aliquot size.

## **20.8 INSTRUMENT DETECTION LIMITS (IDL)**

**20.8.1** The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

**20.8.2** IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

**20.8.3** If IDL is > than the MDL, it may be used as the reported MDL.

## **20.9      VERIFICATION OF DETECTION AND REPORTING LIMITS**

**20.9.1**      Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL for single analyte analyses (e.g., most wet chemistry methods) and 1-4 times the calculated MDL for multiple analyte methods (e.g., GC, GCMS, ICP, etc.). For analyses done in support of the DOD QSM, the spike must be 1-3 times the calculated MDL. The analytes must be qualitatively identified. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

**20.9.2**      When a Reporting limit is initially established, it must be verified by the analysis of a low level standard or QC sample (LCS at 1-2 times the reporting limit). This may be accomplished as part of the MDL determination. If data is not reported below the reporting limit for the method, an annual reporting limit verification sample must be analyzed. This spiked sample must go through the preparation and analysis and meet the acceptance criteria listed in the method SOP.

## **20.10     RETENTION TIME WINDOWS**

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method.

For GC, HPLC and IC methods, there must be sufficient separation between analyte peaks so as to not misidentify analytes. In the mid-level standard, the distance between the valley and peak height cannot be any less than 25% of the sum of the peak heights of the analytes. This also applies to GCMS in the case where the two compounds share the same quantitation ion.

**Note:**      Some analytes do not separate sufficiently to be able to identify or quantitate them as separate analytes (e.g. m-xylene and p-xylene) and are quantitated and reported as a single analyte (e.g. m,p-xylenes).

Once the analyst has determined that the instrument is in optimum working condition through calibration and calibration verification procedures, he or she uses a mid-range calibration or calibration verification standard to establish the retention times for each of the individual analytes in a method. The analyst makes three injections of the same standard over a 72-hour (24 hr period for Method 300.0) period, tabulating the retention times for each analyte for each of the three injections. The width of retention time window is normally the average absolute retention time  $\pm$  3 Standard Deviations. A peak outside the retention time window will not be identified by the computer as a positive match of the analyte of interest.

It is possible for the statistically calculated RT window to be too tight and need to be adjusted based on analyst experience. In these instances method default retention time windows may be used (e.g., for 8000 series methods a default of 0.03 minutes may be used, and EPA CLP 0.05 minutes is used). The same concept is applied when any peak outside of that window will not be

identified by the computer as a positive match. If a default minimum RT window is used it is specified in the analytical SOP.

The calibration verification standard at the beginning of a run may be used to adjust the RT for an analyte. This is essentially re-centering the window but the size of the window remains the same. The RTs are verified when all analytes are within their RT windows and are properly identified.

## **20.11 EVALUATION OF SELECTIVITY**

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, mass resolution, second column confirmation, ICP interelement interference checks, chromatography retention time windows, and sample blanks.

## **20.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT**

**20.12.1** Uncertainty is “a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand” (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result’s validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an “expanded uncertainty”: the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor  $k=2$ .

**20.12.2** Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

**20.12.3** Refer to SOP KNOX-QA-0019 “Estimation of Measurement Uncertainty” for details regarding calculations that may be used to estimate measurement uncertainty associated with laboratory results.

**20.12.4** In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g. 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

## **20.13 CONTROL OF DATA**

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

### **20.13.1 Computer and Electronic Data Related Requirements**



The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running QuantIMS which is an in-house developed LIMS system that has been highly customized to meet the needs of TestAmerica Knoxville. It is referred to as LIMS for the remainder of this section. The LIMS utilizes a DB2 Database which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

**20.13.1.1 Maintain the Database Integrity:** Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

**Note:** “Commercial off-the-shelf software in use within the designed application range is considered to be sufficiently validated.” *From NELAC 2003 Standard.* However, laboratory specific configurations or modifications are validated prior to use.

- In order to assure accuracy, all data entered or transferred into the LIMS data system goes through a minimum of two levels of review.
- The QA department performs random data audits to ensure the correct information has been reported.
- Changes to reports are documented through the use of an audit trail in the LIMS.
- Analytical data file security is provided through three policies.
  - The first policy forbids unauthorized personnel from using laboratory data acquisition computers.
  - The second policy is the implementation of network passwords and login names that restrict directory access.
  - The third layer is maintained through the LIMS and includes the use of username/password combinations to gain access to the LIMS system, the fact that all data in the LIMS is associated with the user who added/reviewed the data, and the restriction of review authority of data.
- All software installations will be in accordance with any relevant copyright licensing regulations.
- All software installed on any computer within the laboratory must be approved by the Information Technology Department regional support technician assigned to the laboratory. Shrink-wrapped or otherwise sealed OEM software that is directly related to instrument usage does not need approval but the Information Technology department must be notified of the installation.
- Anti-virus software shall be installed on all servers and workstations. The anti-virus software shall be configured to check for virus signature file and program updates on a daily basis and these updates will be pushed to all servers and workstations. The anti-virus software will be configured to clean any virus-infected file if possible, otherwise

the file will be deleted. Disks and CDs brought from any outside source that are not OEM software must be scanned for viruses before being accessed.

- **Interlab LIMS Permissions Policy**

- PURPOSE - The purpose of this policy is to provide a mechanism for maintaining the integrity of information contained in each laboratory's LIMS while providing the necessary access for information sharing to staff at other laboratory facilities.
- DEFINITIONS - Host Laboratory: The laboratory facility that 'owns' the LIMS system or 'hosts' a project/job.
- POLICIES
  - (a) All permissions for the laboratory's LIMS system must only be granted by a representative of that laboratory.
    - If someone outside of the host lab needs permissions for Project Management or other uses, they must go through the Lab Director or his/her designated representative.
    - Permissions must never be granted without the knowledge of the host laboratory.
  - (b) Only laboratory analytical or QA staff from the home laboratory may have edit permissions for laboratory analysis data.
  - (c) Any changes made in laboratory's LIMS system:
    - Must be documented and traceable.
    - If made by staff of an affiliate lab, written permission from the home lab to make the changes (email approval is sufficient) is required.
    - No corrections may be made in another laboratory's system without their knowledge.
  - (d) Data qualifiers in laboratory reports must only be corrected, edited, etc. by the staff at the host laboratory.
  - (e) Full analytical data "View" only permissions may be granted to outside Project Management and Sales staff. Search permissions may also be granted so status may be checked.
  - (f) All qualifiers must be approved by QA staff before adding to standard reference tables.
  - (g) Please contact Corporate QA or IT staff if you have any questions regarding implementation or interpretation of this policy.

**20.13.1.2 Ensure Information Availability:** Protection against loss of information or service through scheduled back-ups, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

- Ensured by timely backup procedures on reliable backup media, stable file server network architecture, and UPS protection.
- UPS Protection:
  - Each fileserver is protected by an appropriate power protection/backup unit. In the event of a power outage, there is approximately 15-30 minutes of up-time for the servers prior to shutdown. This allows for proper shutdown procedures to be followed with the fileservers.
- File Server Architecture



- All files are maintained on multiple Windows NT or newer servers which are secured physically in the Information Technology computer room. Access to these servers is limited to members of the Information Technology staff.
- All supporting software is maintained for at least 5 years from the last raw data generated using that software. [Length of time is dependent on local regulations or client requirements (e.g., OVAP requires 10 years). ]
- System Back-up Overview and Procedures
  - Data from both servers and instrument attached PC's are backed up and purged in compliance with the corporate back-up policy.
  - A Maintenance Plan has been defined to create a daily archive of all data within the LIMS database to a backup location. This backup is initiated automatically by either the database or back-up system.
  - Backup tapes will be stored in compliance with the corporate Data Backup Policy. Backup verifications are carried out in accordance with the corporate Data Backup Policy.
  - Instrument data back-ups are verified on a periodic basis by the QA department when performing electronic data audits. These audits include data that has been moved to a back-up location ensuring that it is properly archived.

**20.13.1.3 Maintain Confidentiality:** Ensure data confidentiality through physical access controls, and encryption when electronically transmitting data.

- All servers are located in a secure area of the IT department offices. Access to the servers is limited to IT staff members.
- The company website contains SSL (Secure Socket Layer) encryption for secure website sessions and data transfers.

### **20.13.2 Data Reduction**

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, "*Acceptable Manual Integration Practices*" and as described in KN-QA-002 "Manual Integrations."

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction is not performed unless it is specifically required by the method. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for the methods are summarized in the respective analytical SOPs.

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets.
- Input of raw data for computer processing.
- Direct acquisition and processing of raw data by a computer.

- 20.13.2.1** If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.
- 20.13.2.2** For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.
- 20.13.2.3** If data are directly acquired from instrumentation and processed, the analyst must verify that the following are correct:
- Project and sample numbers
  - Calibration constants/RFs
  - Units
  - Numerical values used for reporting limits.
- 20.13.2.4** In general, sample results are reported to 2 significant figures on the final report. See TestAmerica Knoxville policy QA-004 regarding LIMS significant figure algorithms.

### **20.13.3 Logbook / Worksheet Use Guidelines**

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.).

- Corrections are made following the procedures outlined in Section 13.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"ed out, signed and dated.
- Worksheets are created with the approval of the Technical Director/QA Manager at the facility. The QA Manager or designee controls all worksheets following the procedures in Section 6.

### **20.13.4 Review / Verification Procedures**

A minimum of three levels of review are performed before final sample data is reported to the client.

- First level technical review.
- Second level technical review.
- Third level project management completeness review.

- 20.13.4.1** The initial first level technical review ensures that:
- Sample preparation information is correct and complete including documentation of standard identification, sample amounts, etc.
  - Analysis information is correct and complete including proper identification of

- analysis output (charts, chromatograms, mass spectra, etc.).
- Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers.
- The appropriate SOPs have been followed and are identified in the project records.
- Proper documentation procedures have been followed.
- All nonconformances have been documented.
- Special sample preparation and analytical requirements have been met.
- The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or otherwise specified by the client.

The initial verification of the data reduction (level 1 review) by the analyst is documented on a data review checklist, which is signed and dated by the analyst.

**20.13.4.2** Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through level 1 review is performed by an experienced peer, supervisor, or designee. This check (level 2 review) is performed to ensure that level 1 review has been completed correctly and thoroughly. This review includes an evaluation of all items required in the raw data package. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:

- Adherence of the procedure used to the requested analytical method SOP.
- Correct interpretation of chromatograms, mass spectra, etc.
- Correctness of numerical input when computer programs are used (checked randomly).
- Correct identification and quantitation of constituents with appropriate qualifiers.
- Numerical correctness of calculations and formulas (checked randomly).
- Acceptability of QC data.
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.).
- Documentation of dilution factors, standard concentrations, etc.
- Sample holding time assessment.

The level 2 review also serves as verification that the process the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and specific instructions given on the project QAS or equivalent summary form.
- Nonconforming events have been addressed by corrective action as defined on a nonconformance memo.
- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct.
- The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.

The specific items covered in the level 2 review may vary according to the analytical method, but this review of the data must be documented by signing the data review checklist.

**20.13.4.3** A third-level review is performed by the PM. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG).
- Every parameter or target compound requested is reported with either a value or reporting limit.
- The correct units and correct number of significant figures are utilized.
- All nonconformances and receipt variances with the resolution, including holding time violations and data evaluation statements that impact the data quality, are accompanied by clearly expressed comments from the laboratory.
- The final report is legible, contains all the supporting documentation required by the project, and is in either the standard TestAmerica Knoxville format or in the client-required format.
- Implement checks to monitor the quality of laboratory results using correlation of results for different parameters of a sample (for example, do the TOC results justify the concentration of organic compounds found by GC/MS?)

A narrative to accompany the final report will be finalized by the PM. This narrative will include relevant comments collected during the earlier reviews.

**20.13.4.4** Additional reviews are performed by the QA department. These reviews are typically performed after the data has been reported to the client. These reviews are a routine part of the Quality System and are discussed in further detail in Section 16.2.

#### **20.13.5 Manual Integrations**

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines. Further details on documenting manual integration are found in SOP KN-QA-002, Manual Integrations.

- 20.13.5.1** The analyst must adjust the baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.
- 20.13.5.2** Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.
- 20.13.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- 20.13.5.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

**Figure 20-1.**  
**Example - Demonstration of Capability Documentation**

TestAmerica Knoxville DEMONSTRATION OF CAPABILITY & CERTIFICATION STATEMENT	
Type of Training:	<input type="checkbox"/> Initial <input type="checkbox"/> On-going
Date:	
Laboratory Name:	TestAmerica Knoxville
Laboratory Address:	5815 Middlebrook Pike, Knoxville TN 37921
Analyst(s) Name(s):	Group:
Matrix:	
Method Number/SOP Number/Analyte/Class of Analytes/Measured Parameters:	

- ☐ The analyst(s) listed above has completed four consecutive LCSs with achieved acceptable target analyte recoveries and concentrations within method- and/or laboratory-established control limits. (Project/Batch IDs: \_\_\_\_\_)
- ☐ The analyst(s) listed above has successfully prepared and/or analyzed the following "blind" performance evaluation sample: \_\_\_\_\_.
- ☐ Demonstration of capability (See SOP KNOX-QA-0009) required for initial demonstration. If spiking was not an option, proficiency was demonstrated by: \_\_\_\_\_.

*NOTE: The analyst also certifies by signing this form that he/she has read the SOP(s) indicated above and understands the applicable safety requirements in the SOP(s) indicated above to perform this activity.*

**Analyst:** \_\_\_\_\_ **Date:** \_\_\_\_\_.

**Approved By:** \_\_\_\_\_ **Date:** \_\_\_\_\_.  
(Supervisor/Manager)

#### **Certification Statement**

We, the undersigned, CERTIFY that:

1. The analyst(s) identified above, using the cited test method, which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program and the TestAmerica Knoxville Quality Assurance Program, have met the Demonstration of Capability.
2. The test method(s) was performed by the analyst(s) identified on this certification.
3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.
4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (\*).
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

<u>Robyn M. Wagner</u>	_____	_____
Technical Director's Name	Signature	Date
<u>Christopher W. Rigell</u>	_____	_____
Quality Assurance Manager's Name	Signature	Date

\* True: Consistent with supporting data.

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.

Complete: Includes the results of all supporting performance testing.

Self-explanatory: Data properly labeled and stored so that the results are traceable and require no additional explanation.

QA057R5.doc, 7/10/07

**Figure 20-2. Example - New Method Checklist**

**TestAmerica Knoxville Method Development Checklist**

<b>Project Name:</b>		<b>Project Leader:</b>	
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Method Set-up	Comments
1. Install, Set-up Instruments and Equipment	
2. Define Targets and Target RLs	
3. Describe Interferences (Define method selectivity)	
4. Specify Processing Algorithm/Platform and Applications (Must first define data entities, calculations, output.)	
5. Define Internal Standards (if applicable)	
6. Define Instrument configuration. (Columns, method of introduction)	
7. Define Instrument Conditions/Operating Parameters	
8. Determine Calibration Levels	
9. Design Calibration Series	
10. Draft Analytical SOP	
11. Prepare/Update Logbooks Associated with SOP	
12. Procure Calibration Series Standards	
• Complete receipt log and file vendor certificates	
13. Procure Second Source Standard(s)	
• Complete receipt log and file vendor certificates	
14. Formulate Calibration Series	
• Include standard at or below desired reporting limit	
15. Formulate Second Source Standard	
16. Formulate Surrogate Solution (if applicable)	
17. Formulate Internal Standards (if applicable)	
18. Formulate Spike Solution	
• Set to midpoint or in lower half of calibration range	
19. Obtain Valid Calibration	
20. Validate the Data Processing Method	
21. Analyze Second Source Standards	
22. Define Cleanup Methods (if applicable)	
23. Draft Preparation/Cleanup SOP	
24. Test Surrogate Mix	
25. Test Internal Standard Mix	
26. Test Spike Solution	
27. Update/Review SOPs	
28. Verify Calculation Software (Standard)	
29. Verify Calculation Software (Sample)	
• QuantIMS test lot	
30. Obtain Instrument Detection Limit (if applicable)	
31. Obtain Valid Initial Demonstration of Capability (IDOC, IPR) (for each matrix)	
32. Obtain Valid Method Detection Limit (for each matrix)	
• Evaluate method blanks (check for interferences)	
33. Run MDL Check (for each matrix)	

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QA085R4, 7/20/07

**Table 20-1 Analytical Methods and Matrices**

Analytical Parameters	Fields of Testing <sup>1</sup>				
	Drinking Water (SDWA)	Non-Potable Water (CWA/RCRA)	Solid and Chemical Materials (RCRA-SW846)	Air and Emissions (CAA)	Biological Tissue
Anions (Br <sup>-</sup> , Cl <sup>-</sup> , F <sup>-</sup> , NO <sub>2</sub> , NO <sub>3</sub> , o-PO <sub>4</sub> , SO <sub>4</sub> )	<b>EPA 300.0</b>	<b>EPA 300.0</b> <b>EPA 9056</b>	<b>EPA 9056</b>	Mod Method 26A/0050 (9057 Mod)	---
Ash	---	---	ASTM D482	---	---
Cyanide (Total)	---	<b>EPA 9012A</b> EPA 335.4	<b>EPA 9012A</b>	---	---
Density	---	---	ASTM D1963, D854	---	---
Halogens	---	---	EPA 5050/9056 (KNOX-WC-0016)	---	---
Heat of Combustion (Btu)	---	---	ASTM D5865, D240	---	---
Hexavalent Chromium	---	---	---	Method 0061/7199	---
Perchlorate	<b>EPA 314.0</b>	EPA 314.0		---	---
pH	<b>EPA 150.1</b>	<b>EPA 9040B</b> SM 4500-H+B	<b>EPA 9040B</b> <b>EPA 9045C</b>	---	---
Hardness	SM 2340B	<b>SM 2340B</b>	---	---	---
Particulates	---	---	---	PM-10, 40CFR, Part 50, App. J & App. B Method 0050/5 40CFR Part 60 App. A	---
Mercury	<b>EPA 245.1</b>	<b>EPA 245.1</b> <b>EPA 7470A</b>	<b>EPA 7470A</b> <b>EPA 7471A</b>	<b>EPA 0060/7470A</b> ASTM D6784-02 EPA IO-3.1/7470A <b>EPA Method 29/7470A</b>	EPA 7471A
Metals	<b>EPA 200.7</b>	<b>EPA 200.7</b> <b>EPA 6010B</b>	<b>EPA 6010B</b>	<b>EPA 0060/6010B</b> EPA IO-3.1/6010B <b>EPA Method 29/6010B</b> 40CFR60-12 (Pb)	EPA 6010B
Sulfur			ASTM D3177, ASTM D129		
Toxicity Characteristic Leaching Procedure (TCLP) & Synthetic Precipitation Leaching (SPLP)	---	---	<b>EPA 1311</b> <b>EPA 1312</b>	---	---
Viscosity	---	---	ASTM D445	---	---



**Table 20-1 (continued) Analytical Methods and Matrices**

Analytical Parameters	Fields of Testing <sup>1</sup>				
	Drinking Water (SDWA)	Non-Potable Water (CWA/RCRA)	Solid and Chemical Materials (RCRA-SW846)	Air and Emissions (CAA)	Biological Tissue
Volatiles by GC/MS	---	<b>EPA 8260B</b>	<b>EPA 8260B</b>	TO-14A, TO-15  <b>EPA 0031/5041A (VOST)</b>	---
Volatiles by GC	---	---	---	EPA 0040 Condensates ASTM D1946	---
Semivolatiles by GC/MS	---	<b>EPA 8270C</b>	<b>EPA 8270C</b>	<b>TO-13A</b> <b>EPA 3542/8270C</b>	---
Semivolatiles by GC	---	<b>Pesticides EPA 8081A, PCBs EPA 8082, Congeners EPA 8082</b>	<b>Pesticides EPA 8081A, NOAA/8081A PCBs EPA 8082, Congeners EPA 8082</b>	<b>TO-4A, TCO/Grav</b>	PCBs EPA 8082, <b>Congeners EPA 8082</b> , Pesticides NOAA/8081A
Explosives by HPLC	---	<b>EPA 8330</b> <b>EPA 8332</b>	<b>EPA 8330</b> <b>EPA 8332</b>	---	---
Dioxins/Furans by HRGC/HRMS	<b>EPA 1613B</b>	<b>EPA 1613B</b> <b>EPA 8290</b>	<b>EPA 8290</b>	<b>TO-9A, Method 23/8290</b> <b>EPA 0023A/8290</b>	<b>EPA 8290</b> <b>EPA 1613B</b>
Lipids					EPA 8290, Micro-Lipids (KNOX-OP-0019)
PCBs by HRGC/HRMS	---	<b>EPA 1668A</b>	<b>EPA 1668A</b>	EPA 1668A	<b>EPA 1668A</b>
Dioxins/Furans by Immunoassay Screen	---	---	EPA 4000, EPA 4025 Mod	---	---
PCBs by Immunoassay Screen	---	---	EPA 4000	---	---
Polycyclic Aromatic Hydrocarbons (PAH)	---	---	NOAA/8270C (PAHs and Alkyl PAHs)	KNOX-ID-0016 (CARB 429 Mod, HRGC/LRMS)	NOAA/8270C (PAHs and Alkyl PAHs), KNOX-ID-0016 (HRGC/LRMS)

<sup>(1)</sup> NELAC accredited methods are highlighted in bold font.

## **SECTION 21**

### **EQUIPMENT (AND CALIBRATIONS)** **(NELAC 5.5.5)**

#### **21.1      OVERVIEW**

TestAmerica purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs for the analytical methods. A list of laboratory equipment and instrumentation is presented in Table 21-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

#### **21.2      PREVENTIVE MAINTENANCE**

**21.2.1**      TestAmerica Knoxville follows a well-defined program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

**21.2.2**      Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

**21.2.2.1**      Calibrations, routine maintenance, and adjustments are part of the analysts' and Group Leaders' responsibilities. However, service contracts may be in place for some instruments to cover any major repairs.

**21.2.2.2**      High purity gases, reagents, and spare parts are kept on hand to minimize repair time and optimize instrument performance.

**21.2.3**      Table 21-2 summarizes the schedule for routine maintenance. It is the responsibility of each Group Leader to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to document maintenance is also used to monitor performance.)

**21.2.4**      Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all

major pieces of equipment. Instrument maintenance logs may also be used to specify instrument operating parameters.

**21.2.4.1** Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

**21.2.4.2** Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control, e.g., CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.). An entry in the associated analytical runlog documenting successful calibration may also be used to document instrument return to control following maintenance.

**21.2.4.3** When maintenance or repair is performed by an outside agency, service receipts detailing the service performed are affixed into the maintenance logbook. The analyst signs across the entered item and the logbook page so that it is clear that the insert is missing if only half a signature is found in the logbook.

**21.2.5** In addition, the maintenance records contain:

- The identification of the instrument/equipment (instrument's Serial Number and Model Number).
- The date the instrument/equipment was put into use.
- If available, the condition when the instrument was received (e.g., new, used, reconditioned).
- Refer to Table 21-2 for preventative maintenance procedures.

**21.2.6** If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits), it shall be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses (refer to Sections 12 and 13).

**21.2.7** In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted using the procedures outlined in Section 8.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

### **21.3      SUPPORT EQUIPMENT**

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

#### **21.3.1      Weights and Balances**

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if the laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Further details on balance calibration may be found in SOP KNOX-QA-0005, Balance Calibration and Weight Verification.

#### **21.3.2      pH, Conductivity, and Turbidity Meters**

The pH meters used in the laboratory are accurate to  $\pm 0.1$  pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

#### **21.3.3      Thermometers**

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. Digital thermometers are calibrated quarterly.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer has increments of 0.2 °C, and has a range applicable to all method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

Thermometer calibrations are documented as described in SOP KNOX-QA-0010, Temperature Monitoring and Thermometer Calibration.

#### **21.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators**

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day. Monitoring is performed 7 days per week to ensure that samples remain within an acceptable range. This monitoring of sample storage is done using max/min thermometers.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between 4 +/-2°C.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

The temperatures are documented in temperature logbooks or in the method-specific logbooks/benchsheets.

#### **21.3.5 Autopipettors, Dilutors, and Syringes**

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are checked for accuracy at least quarterly. Glass micro-syringes are considered the same as Class A glassware.

The laboratory maintains a sufficient inventory of autopipettors, and dilutors of differing capacities that fulfill all method requirements.

These devices are given unique identification numbers, and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

Any device not regularly verified can not be used for any quantitative measurements.

Refer to SOP KNOX-QA-0012, Pipetter and Volumetric Labware Calibration Verification.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

## **21.4      INSTRUMENT CALIBRATIONS**

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration).

Sample results must be quantitated from the initial calibration and must not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 13).

**Note:** Instruments are calibrated initially and as needed after that and at least annually. The annual initial calibration requirement does not apply to isotope dilution methods.

### **21.4.1      CALIBRATION STANDARDS**

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. However, the general procedures are described below.

**21.4.1.1** For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods. If a reference or mandated method does not specify the number of calibration standards, the minimum number is three, not including blanks or a zero standard. All of the standard solutions are prepared using Class A volumetric glassware, calibrated pipettes, and/or microsyringes and appropriate laboratory quality solvents and stock standards.

**21.4.1.2** Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to NIST whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. A standard log and a certificate file is maintained for each department, containing concentration, date of

receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.

- 21.4.1.3** The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).
- 21.4.1.4** The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The lowest calibration standard must be at or below the reporting limit. An exception to this rule is for ICP methods where the low level standard is analyzed after calibration; see Section 21.4.3.
- 21.4.1.5** Given the number of target compounds addressed by some of the organic methods, it may be necessary to prepare several sets of calibration standards, each set consisting of the appropriate number of solutions at different concentrations. The initial calibration will then involve the analysis of each of these sets of the appropriate number of standards.
- 21.4.1.6** All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a working standard made by a different analyst may be used for verification. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

#### **21.4.2 CALIBRATION FOR ORGANIC METHODS (GC, HPLC, GC/MS)**

- 21.4.2.1** Many of the organic analytical methods utilize an internal standard calibration (GCMS and some GC). Because of the complex nature of the multipeak chromatograms produced by the method, some instruments necessitate the use of external standard calibration (most GC and HPLC). Surrogate compounds are included in the calibration processes for all appropriate organic analyses. For more details on the calibration types listed below, refer to SOP No. CA-Q-S-005, Calibration Curves.
- 21.4.2.2** Once the operating parameters have been established according to the method, each instrument is calibrated for the appropriate method. The analyst prepares five or more standard solutions at various concentrations containing all of the analytes of interest, internal standards, and surrogates that are appropriate for the method. Note: There are a several EPA methods that have different requirements and are exceptions (e.g. EPA 547) where a minimum of 3 calibration standards are prepared and analyzed.



**21.4.2.3** The standard solutions are introduced into the instrument in the same manner as samples are; whether it be by direct injection, by headspace analysis, or by purge and trap. The calibration factor (CF) for methods that use external standards, and the response factor (RF) for methods that use internal standards are calculated for the five standards.

- External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the response to the amount of analyte in the calibration standard is defined as the Calibration factor (CF).
- Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. The use of specific internal standards is available in the method SOP.

Whichever internal standards are employed, the analyst needs to demonstrate that the measurement of the internal standard is not affected by method analytes and surrogates or by matrix interferences. In general, internal standard calibration is not as useful for GC and HPLC methods with non-MS detectors because of the inability to chromatographically resolve many internal standards from the target compounds. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

When preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes will vary. The internal standard solution will contain one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution (e.g., not all internal standards need to be at the same concentration in this solution). The mass of each internal standard added to each sample extract immediately prior to injection into the instrument or to each sample prior to purging must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts should be such that minimal dilution of the extract occurs (e.g., 10  $\mu$ L of solution added to a 1 mL final extract results in only a negligible 1% change in the final extract volume which can be ignored in the calculations).

An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard should produce an instrument response (e.g.,



area counts) that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This should result in a minimum response factor of approximately 0.01 for the least responsive target compound. Refer to SOP No. CA-Q-S-005, Calibration Curves, for specific calculations.

**21.4.2.4** Policies regarding the use of calibration standard results for creating the calibration curve are as follows:

- A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting level must be elevated to be the lowest calibration standard used for calibration.
- The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.
- Mid-calibration standards may not be excluded unless an obvious reason is found, i.e., cracked vial, incorrectly made, etc. The failed standard must be re-run immediately and inserted into the initial calibration. If not useful, recalibration is required.

#### **21.4.2.5 Percent RSD Corrective Action**

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

**21.4.2.5.1** The first step is generally to check the instrument operating conditions. This option will apply in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance, changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.

**21.4.2.5.2** If the RSD for any analyte is greater than the applicable acceptance criteria in the applicable analytical SOP, the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

**21.4.2.5.3** A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

**Note:** When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the method quantitation limit is at least as low as the regulatory limit or action level.

**21.4.2.6** Alternatively, the least squares regression may be used to determine linearity. A five point line must result in a correlation coefficient ( $r$ ) of 0.990 or better using the least squares method to be considered acceptable. In many cases it may be preferred that the curves be forced through zero (not to be confused with including the origin as an additional data point, which is not allowed). **Note:** EPA method 8000B does not allow forcing through zero, however the agency has reevaluated this position and has since changed this stance to allow forcing through zero. In addition, from EPA Method 8000C: "However, the use of a linear regression or forcing the regression through zero may NOT be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards." Ohio EPA VAP does not allow forcing through zero.

**21.4.2.7** Instead of a linear curve model (either Average RF or least squares regression), a second order curve (Quadratic) may be used (and preferred) as long as it contains at least six data points. As a rule of thumb, if there is a consistent trend

in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (COD or  $r^2$ ) for the quadratic curve must be at least 0.99 for it to be considered acceptable. For more details on the calculations see Calibration Curve SOP CA-Q-S-005. Some limitations on the use of Quadratic Curve fits:

**21.4.2.7.1** Care **MUST** be exercised to assure that the results from this equation are real, positive, and fit the range of the initial calibration.

**21.4.2.7.2** They **must not** be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).

**21.4.2.7.3** They **must not** be used to compensate for detector saturation. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve and try a 1<sup>st</sup> order fit or average RF.

Note: For methods done in support of the Ohio EPA VAP, quadratic curve fitting may only be done if the compound has historically exhibited a non-linear response. Ohio EPA VAP does not allow forcing through zero.

### **21.4.3     Calibration for Inorganic Analyses**

EPA Method 7000 from EPA SW-846 is a general introduction to the quality control requirements for metals analysis. For inorganic methods, quality control measures set out in the individual methods and in the *Standard Methods for the Examination of Water and Wastewater* (20th Edition) may also be included. The quality control measures used by the laboratory are specified in the analytical Standard Operating Procedures.

In general, inorganic instrumentation is calibrated with external standards. An exception would be Inductively Coupled Plasma (ICP). These analyses may use an internal standard to compensate for viscosity or other matrix effects. While the calibration procedures are much the same for inorganics as they are for organics, CF's or RF's are not used. The calibration model in 21.4.2.6 is generally used for most methods, however in some instances the model from section 21.4.2.7 may be used. A correlation coefficient ( $r$ ) of 0.995 or greater must be used to accept a calibration curve generated for an inorganic procedure. Correlation coefficients are determined by computer software and documented as part of the calibration raw data. Coefficients of calibration curves used for quantitation must be documented as part of the raw data. Curves are not allowed to be stored in calculator memories and must be written on the raw data for the purposes of data validation.

**21.4.3.1** "Calibrations" for titrimetric analyses are performed by standardizing the titrants against a primary standard solution. See specific methods in *Standard Methods for the Examination of Water and Wastewater* (20th Edition) for more information.

**21.4.3.2** Spreadsheets that are used for general chemistry calculations must have all cells containing calculations locked to prevent accidental changes to the calculations.

**21.4.3.3** Instrument technologies (e.g. ICP) with validated techniques from the instrument manufacturer or other methods using a zero point and single point calibration require the following:

**21.4.3.3.1** The instrument is calibrated using a zero point and a single point calibration standard.

**21.4.3.3.2** The linear range is established by analyzing a series of standards to determine the upper limit of the linear range.

**21.4.3.3.3** Sample results within the established linear range do not need to be qualified.

**21.4.3.3.4** The zero point and single standard is run daily with each analytical sequence.

**21.4.3.3.5** A standard at the RL is analyzed daily with each analytical sequence and must meet established acceptance criteria.

**21.4.3.3.6** The linearity is verified at a frequency established by the manufacturer or method. Refer to SOP KNOX-MT-0007, ICP AES for further details.

#### **21.4.4**     **Calibration Verification**

The calibration relationship established during the initial calibration must be verified at periodic intervals as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

**Note:**     The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration, and is not appropriate nor permitted in SW-846 chromatographic procedures for trace environmental analyses.

**21.4.4.1**     Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications.) The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.

**21.4.4.2**     A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements; see specific SOPs. Most Inorganic methods require the CCV to be analyzed after every 10 samples.

**21.4.4.3**     The acceptance limits for calibration verifications can be found in each method SOP. As a rule of thumb: GCMS  $\pm$  20%, GC and HPLC  $\pm$  15%, Inorganics:  $\pm$  10 or 15%. Actual methods may have wider or tighter limits; see the analytical method SOPs for specifics.

**21.4.4.4** If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF, or RF values from the initial calibration to quantitate sample results.

**21.4.4.5** If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new initial instrument calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

**21.4.4.5.1** When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

**21.4.4.5.2** When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

#### **21.4.4.6 Verification of Linear Calibrations**

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

The Percent Difference is calculated as follows:

$$\% \text{ Difference} = \frac{(\text{CF(v) or RF(v)}) - (\text{Avg. CF or RF})}{(\text{Avg. CF or RF})} \times 100$$

Where: CF(v) or RF(v) = CF or RF from verification standard  
Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

$$\% \text{ Drift} = \frac{\text{Result} - \text{True Value}}{\text{True Value}} \times 100$$

The Percent Recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100$$

#### **21.4.4.7 Verification of a Non-Linear Calibration**

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations described in 21.4.4.6 above.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The required frequency is found in the analytical SOPs.

**Note:** If an internal standard calibration is being used (basically GCMS and some GC methods) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

#### **21.5 POLICY ON TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS**

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it will not be reported as a TIC. If the compound is reported on the same form as true TICs, it must be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control)

reported compared to a known standard that is in control (where applicable). TestAmerica Knoxville will provide this service upon client request.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. See SOP KNOX-MS-0014, Tentatively Identified Compounds, for guidelines for making tentative identifications.

## **21.6      POLICY ON GC/MS TUNING**

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

**21.6.1**      The concentration of the BFB or DFTPP must be at or below the concentrations that are referenced in the analytical methods. Part of the purpose of the tune is to demonstrate sensitivity and analyzing solutions at higher concentrations does not support this purpose. Tune failures may be due to saturation and a lower BFB/DFTPP concentration may be warranted.

**21.6.2**      Tune evaluations usually utilize the "Autofind" function and are set up to look at the apex +/- 1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction cannot include any part of the target peak.

### **21.6.3      Other Options or if Auto Tune Fails:**

**21.6.3.1**      Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. In this case, manually identify and average the apex peak +/- 1 scan and background correct as in 21.6.2 above. This is consistent with EPA 8260 and 8270.

**21.6.3.2**      Or the scan across the peak at one half peak height may be averaged and background corrected. This is consistent with Standard Methods 6200, EPA 624 and EPA 625.

**21.6.3.3**      Adjustments such as adjustments to the repeller and ion focus lenses, adjusting the EM Voltage, etc. may be made prior to tune verification as long as all of the subsequent injections in the 12 hour tune cycle are analyzed under the same MS tune settings and it is documented in the run sequence log and/or maintenance log that an adjustment was made. Excessive adjusting (more than 2 tries) without clear documentation is not allowed. Necessary maintenance is performed and documented in the instrument log.



**21.6.3.4** A single scan at the Apex (only) may also be used for the evaluation of the tune. For SW 846 and EPA 600 series methods, background correction is still required.

**21.6.3.5** Cleaning the source or other maintenance may be performed and then follow steps for tune evaluation above. Note: If significant maintenance was performed, see methods 8000B or 8000C, then the instrument may require recalibration prior to proceeding.

**21.6.4** Tune evaluation printouts must include the chromatogram and spectra as well as the Tune evaluation information. In addition, the verifications must be sent directly to the printer or pdf file (no screen prints for DFTPP or BFB tunes). This ability must be built into the instrument or data processing software.

**21.6.5** All MS tune settings must remain constant between running the tune check and all other samples. It is recommended that a separate tune method not be used, however a separate method may be used as long as the MS conditions between the methods are the same as the sample analysis method and tracked so any changes that are made to the analysis method are also made to the tune method.



**Table 21-1. Laboratory Instrument List**

<b>Instrument Type</b>	<b>Manufacturer</b>	<b>Model Number</b>	<b>Serial Number</b>	<b>Year Put into Service</b>	<b>Condition When Received</b>
ICP-AES	Thermo Jarrell Ash	61E Trace	512990	2000	NEW
ICP-AES	Thermo Jarrell Ash	61E Trace	248490	1994	NEW
Mercury Analyzer (CVAAS)	Leeman Labs, Inc.	Hydra AA	62434	2001	NEW
GC/MS Semivolatiles	Hewlett-Packard	5973/6890	US82321805	1999	NEW
GC/MS Semivolatiles	Agilent Technologies	5973/6890	US10451834	2001	NEW
GC/MS Volatiles	Hewlett-Packard	5972A/6890	3435A01974	1999	NEW
GC/MS Volatiles	Agilent Technologies	5973/6890	US10129076	2001	NEW
GC/MS Volatiles	Agilent Technologies	5973/6890	US44621247	2005	NEW
GC/MS Volatiles	Agilent Technologies	5973/6890N	US10345111	2003	NEW
GC/MS Volatiles	Hewlett-Packard	5973/6890	US80310976	1998	NEW
GC/MS Volatiles	Hewlett-Packard	5973/6890	US80210991	1998	NEW
GC/MS Volatiles	Hewlett-Packard	5973/6890	US91911868	2000	NEW
HRGC/HRMS	Finnigan	MAT-95/6890	2631	1991	NEW
HRGC/HRMS	Finnigan	MAT-90S/6890	US00001760	2001	USED
HRGC/HRMS	Finnigan	MAT-95S/6890	2725	1998	NEW
HRGC/LRMS	Hewlett-Packard	5973/6890	US94260093	2000	NEW
Combiflash Separation Chromatograph	Isco	Combi Flash Sq 1600	212287 (pump)	2001	NEW
Ion Chromatograph	Dionex	DX-320	00070167	2000	NEW
Ion Chromatograph	Dionex	DX-600	02070458	2002	NEW
Ion Chromatograph	Dionex	ICS-1500	03100244	2003	NEW
Flow Injection Spectrophotometer	Lachat	Quick Chem 8000	A83000-2135	2003	NEW
Calorimeter	Parr	Bomb Calorimeter 1266	A1126DDEA	2003	NEW
Furnace (Ash)	Barnstead Thermolyne Furnace	30400	1262041236063	2003	NEW
GPC	J2 Scientific	GPC Accuprep	05L-1179-4.0	2003	NEW
pH Meter	Accumet	Model 15, pH Meter	C0025140	1998	NEW
Spectrophotometer	Thermo Electron	Spectronic 20D+	3DUK270002	2007	NEW
GC-FID	Varian	3600 FID	0295	1998	NEW
GC-FID	Varian	3300 FID	3890	1987	NEW
GC-TCD	Varian	3400 TCD	3961	1989	NEW
GC-ECD	Agilent Technologies	6890N ECD	CN10615005	2006	NEW
GC-FID	Varian	3400 FID	4644	1987	NEW
GC-ECD	Hewlett-Packard	6890 ECD	US00021272	1997	NEW
GC-ECD	Agilent Technologies	6890 ECD	US00041050	2001	NEW
GC-ECD	Hewlett-Packard	5890 ECD	2618A07840	1986	NEW
GC-ECD	Agilent Technologies	6890 ECD	US10346012	2003	NEW
HPLC	Hewlett-Packard	Series 1100	US80303183	1999	NEW
HPLC	Hewlett-Packard	Series 1100	DE11113404	1998	NEW
HPLC	Agilent	Series 1200	DE62962744	2007	NEW

Table 21-2 – Preventive Maintenance Procedures for Laboratory Equipment		
Instrument/ Equipment Type	Activity	Frequency
Gas chromatograph	Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Daily – documented in runlog
	Fill solvent reservoirs	Daily – documented in runlog
	Check temperature program/instrument method.	Daily – documented in runlog
	Air analyses: Check liquid nitrogen and carrier gas levels.	Daily – documented in runlog
	Break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.	As needed - documented in the instrument maintenance log
	Replace injection port liner when front portion of capillary column is removed or when breakdown exceeds limits.	As needed - documented in the instrument maintenance log
	Replace or repair flow controller if constant gas flow cannot be maintained.	As needed - documented in the instrument maintenance log
	Replace fuse.	As needed - documented in the instrument maintenance log
	Reactivate external carrier gas dryers.	As needed - documented in the instrument maintenance log
	Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace igniter. ECD: follow manufacturers suggested maintenance schedule	As needed - documented in the instrument maintenance log
	Check inlets, septa. Replace septum.	As needed - documented in the instrument maintenance log
	Clean injector port.	As needed - documented in the instrument maintenance log
	Clip column leader.	As needed - documented in the instrument maintenance log
	Reactivate flow controller filter dryers when presence of moisture is suspected.	As needed - documented in the instrument maintenance log
	ECD: perform wipe test.	Semiannually – document in the instrument maintenance log
Ion Chromatograph	Check all lines for crimping, leaks and discoloration.	Daily – documented in runlog
	Check pump and gas pressure.	Daily – documented in runlog

Table 21-2 – Preventive Maintenance Procedures for Laboratory Equipment		
Instrument/ Equipment Type	Activity	Frequency
	Clean conductivity cell.	As needed - documented in the instrument maintenance log
	Change column and guard column.	As needed - documented in the instrument maintenance log
	Change column and/or guard column bed support	As needed - documented in the instrument maintenance log
	De-gas pump head when flow is erratic.	As needed - documented in the instrument maintenance log
	Check/replace eluant end line filter.	As needed - documented in the instrument maintenance log
HPLC	Empty deionized water reservoir and refill with fresh deionized water. Add solvents to other reservoirs.	Daily – documented in runlog
	Check system pressure.	Daily – documented in runlog
	Check solvent flow and column temperature.	Daily – documented in runlog
	Check proper solvent mix for mobile phase.	Daily – documented in runlog
	Check system for leaks.	Daily – documented in runlog
	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.	As needed - documented in the instrument maintenance log
	Change pump seals when flow becomes inconsistent.	As needed - documented in the instrument maintenance log
	Replace UV lamp.	As needed - documented in the instrument maintenance log
	Replace active inlet valve.	As needed - documented in the instrument maintenance log
	Replace pre-column.	As needed - documented in the instrument maintenance log
	Replace in-line filters.	As needed - documented in the instrument maintenance log
GC/MS (Non Isotope Dilution)	Check for correct column flow and/or inlet pressure.	Daily – documented with successful tune and calibration
	Check temperatures of injector, detector. Verify temperature programs.	Daily – documented with successful tune and calibration
	Check inlets, septa	Daily – documented with successful tune and calibration
	Check baseline level.	Daily – documented with successful tune and calibration
	Check relative abundance and mass assignments of the tuning compounds	Daily – documented with successful tune and calibration

Table 21-2 – Preventive Maintenance Procedures for Laboratory Equipment		
Instrument/ Equipment Type	Activity	Frequency
	Ensure communication is established between the mass spec and the GC system.	Daily – documented with successful tune and calibration
	Check mass calibration (PFTBA or FC-43)	As needed - documented in the instrument maintenance log
	Change injection port liner.	As needed - documented in the instrument maintenance log
	Clip column	As needed - documented in the instrument maintenance log
	Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required level	As needed - documented in the instrument maintenance log
	Clean source, including lenses – the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination	As needed - documented in the instrument maintenance log
	Replace filaments when both filaments burn out or performance indicates need for replacement	As needed - documented in the instrument maintenance log
	Check ion source and analyzer (clean, replace parts as needed)	As needed - documented in the instrument maintenance log
	Clean air filters on GC/MS system.	As needed - documented in the instrument maintenance log
	Replace traps on Entech and Tekmar	As needed - documented in the instrument maintenance log
	Replace side plate o-ring.	As needed - documented in the instrument maintenance log
	Change inlet liners, seals and septa as needed, usually indicated by a loss of response in ana-lytes that are sensitive to active sites or dirty parts	As needed - documented in the instrument maintenance log
	Check calibration vial.	As needed - documented in the instrument maintenance log
	Replace the exhaust filters on the mechanical rough pump every 1-2 years	As needed - documented in the instrument maintenance log
	Replace the diffusion pump fluid.	As needed - documented in the instrument maintenance log
	Replace the split vent trap between injection port and EPC	As needed - documented in the instrument maintenance log
	Change oil in the mechanical rough pump	Semi-annually: documented in the instrument maintenance log
ICP	Check that argon manifold gas pressure is 80 psi.	Daily – documented on chart cover page

Table 21-2 – Preventive Maintenance Procedures for Laboratory Equipment		
Instrument/ Equipment Type	Activity	Frequency
	Check that nebulizer is not clogged.	Daily – documented on chart cover page
	Check that capillary tubing is clean and in good condition.	Daily – documented on chart cover page
	Check that peristaltic pump windings are secure.	Daily – documented on chart cover page
	Check that high voltage switch is on.	Daily – documented on chart cover page
	Check that exhaust fans are working.	Daily – documented on chart cover page
	Clean plasma torch assembly to remove accumulated deposits.	Daily – documented on chart cover page
	Check spray chamber O-rings	Daily – documented on chart cover page
	Clean nebulizer and drain chamber.	As needed
	Clean filters on back of power unit to remove dust.	As needed
	Replace when needed: peristaltic pump tubing sample capillary tubing autosampler sipper probe.	As needed
	Clean and lubricate autosampler arm.	As needed
	Check that cooling water supply system is full and drain bottle is not full.	As needed
	Clean air filter on water cooling system.	As needed
	Manufacturer service engineer for scheduled preventive maintenance service.	Annually
	Change vacuum pump oil on ST1	Annually
Cold Vapor Atomic Absorption (Leeman Mercury Analyzer)	Check tubing.	Daily
	Check maintenance schedule flags.	Daily
	Adjust/change Hg lamp.	As needed
	Clean or replace optical cell.	As needed
	Lubricate pump and autosampler arm.	As needed
	Change the drying tube.	As needed
LACHAT Auto Analyzer	Wipe rollers with isopropanol and apply silicone spray.	As needed

Table 21-2 – Preventive Maintenance Procedures for Laboratory Equipment		
Instrument/ Equipment Type	Activity	Frequency
	Wipe the carriages clean.	As needed
	Replace the tubing, clean port valves and replace the o-rings.	As needed
	Check pump tubing.	Daily
	Check for leaks.	Daily
	Flush manifolds with deionized water after each use.	Daily
	If analyzing for NO <sub>3</sub> / NO <sub>2</sub> , ensure analytical column is functional.	Daily
	Clean pump	Semi-annually
GCMS HRGC/LRMS Isotope Dilution	Replace septum	As needed
	Replace injection port liner	As needed
	Perform column maintenance	As needed
	Replace carrier gas filter	As needed
	Check carrier gas pressure (record)	As needed
	Install column (type and ID No.)	As needed
	Clean ion source	As needed
	Install clean ion volume	As needed
	Install new filament	As needed
	Change mechanical pump oil	Annually
	Check PCB seating and alignment.	As needed
	Check power supply voltages	As needed
GCMS HRGC/HRMS Isotope Dilution	Replace septum	As needed
	Replace injection port liner	As needed
	Perform column maintenance	As needed
	Replace carrier gas filter	As needed
	Check carrier gas pressure (record)	Weekly
	Install column (type and ID No.)	As needed
	Evaluate air spectrum, masses 28,32	Weekly
	Check tray chiller and water level.	Monthly
	Replace inlet seal.	As needed
	Clean injection port body.	As needed
	Replace interface ferrule on xfer line.	As needed
	Clean ion source	As needed
	Install clean ion volume	As needed
	Install new filament	As needed
	Replace PFK reservoir septum (HRMS)	As needed
	Change mechanical pump oil	Annually
	Check coolant level and temp	As needed
	Check cooling fans	Weekly
	Check PCB seating and alignment	Biannually
	Check power supply voltages	As needed

Table 21-2 – Preventive Maintenance Procedures for Laboratory Equipment		
Instrument/ Equipment Type	Activity	Frequency
	Check 5 volt card cage power supply	Monthly
Sonicator	Tune sonicator assembly	Daily
	Disassemble and clean sonicator probe tips.	As Needed
	Replace probe tip.	As Needed
Calorimeter	Check O <sub>2</sub> tank level and change tank if low.	Daily – documented with successful calibration
	Verify temperature with external thermometer if method performance criteria cannot be consistently met.	As Needed
	Replace defective or worn parts and stems on bombs.	As Needed
	Polish bomb interior.	As Needed
Gas Chromatograph (ACS group)	Check for correct column flow and/or inlet pressure	Daily
	Check temperatures of injector, detector.	Daily
	Verify temperature programs.	Daily
	Check baseline level (offset)	Daily
	Replace sparger septum in PAT used for Method 0040	Weekly
	Replace the trap on the purge-and trap.	As Needed
	For Method 0040: Replace the carrier gas and purge gas filters.	As Needed
	Clip column ends and reinstall column	As Needed
	Replace column.	As Needed
	Clean sparger for Method 0040.	As Needed
	Re-ignite FID	As Needed
Muffle Furnace (ACS group)	Verify temperatures with platinum resistance thermometer per SOP.	Annually

**Table 21-3. Periodic Calibration**

<b>Instrument/ Device</b>	<b>Type of Calibration/ Number of Standards</b>	<b>Frequency</b>	<b>Acceptance Limits</b>	<b>Corrective Action</b>
Analytical Balance	Accuracy determined using NIST Type I traceable weights.  Minimum of 2 standards bracketing the weight of interest.	Each day of use	Varies by balance and weight. See Balance SOP.	Clean, check level, recheck. If fails, call QA.
Top Loading Balance	Accuracy determined using A2LA-accredited NIST weights.  Minimum of 2 standards bracketing the weight of interest.	Each day of use	Varies by balance. See Balance SOP.	Clean, check level, recheck. If fails, call QA.
NIST Class 1 Traceable Working Weights	Accuracy verified against NIST Class 1 Reference Weights	Annually	See Balance and Weights SOP	Replace
NIST Class 1 Reference Weights	Accuracy determined by A2LA or NVLAP accredited weights and measurement laboratory.	5 years	As per certificate.	Replace.
NIST- Traceable Reference Thermometer	Accuracy determined by approved calibration laboratory.	5 years	As per certificate.	Replace.
Liquid in Glass Working Thermometer	Against NIST-traceable thermometer	When received and annually at appropriate temperature range for intended use	+/-0.5°C	Replace
Digital Thermometers	Against NIST-traceable thermometer	Quarterly	+/-0.5°C	Replace
Refrigerator	Temperature checked using NIST-traceable thermometer.	Daily.	4 ± 2°C	Check temperature, reread, issue nonconformance, tag out, and move samples to working unit as described in SOP KNOX-QA- 0010 Temperature Monitoring and Thermometer Calibration.



<b>Instrument/ Device</b>	<b>Type of Calibration/ Number of Standards</b>	<b>Frequency</b>	<b>Acceptance Limits</b>	<b>Corrective Action</b>
Freezer	Temperature checked using NIST-traceable thermometer	Daily.	-10 to -20°C <-20°C for Sediment and Tissue Samples	Check temperature, reread, issue nonconformance, tag out, and move samples to working unit as described in SOP KNOX-QA-0010 Temperature Monitoring and Thermometer Calibration.
Oven	Temperature checked using NIST-traceable thermometer.	When in use.	104 ± 1°C (% moisture)	Adjust. Repair or Replace.
Volumetric Dispensing Devices (Eppendorf ® pipette, automatic volumetric or dispensing devices)	One delivery by weight. Using DI water, dispense into tared vessel. Record weight with device ID number. See SOP KNOX-QA-0012 for further details.	Quarterly	± 3%	Adjust. Repair or Replace.
Volumetric Labware (Non-Class A) Used for measuring initial sample volume or final extract or digestate volume.	Verified by weight of DI water. See SOP KNOX-QA-0012 for further details	Once Per New Lot of Volumetric Labware	± 3%	Replace.

## SECTION 22

### MEASUREMENT TRACEABILITY (NELAC 5.5.6)

#### 22.1 OVERVIEW

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. The following definitions are provided by the American Association for Laboratory Accreditation (A2LA):

“Traceability is the property of a measurement result whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, each step in the chain having stated uncertainties.” There are six essential elements:

- An unbroken chain of comparison
- A calculated measurement uncertainty for each step in the chain to allow for an overall uncertainty calculation
- Documentation of each step in each calibration report
- All steps in the chain are performed by individuals with evidence of technical competence and accredited by a recognized accreditation body
- Reference to International Standard (SI) units
- Recalibration at appropriate intervals to preserve traceability

Calibration is defined as “determining and documenting the deviation of the indication of a measuring instrument (or the stated value of a material measure) from the conventional ‘true’ value of the measurand.”

Uncertainty is defined as “a parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measurand.” Measurement of Uncertainty is discussed in Section 20 of this QA Manual.

## **22.2      NIST-TRACEABLE WEIGHTS AND THERMOMETERS**

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and freezers are checked on each day of use.

## **22.3      REFERENCE STANDARDS / MATERIALS**

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, or NVLAP where possible with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. (Refer to Section 9 for additional information on purchasing.) The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst is acceptable for this comparison. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g., calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to Table 9-1 in Section 9 for general storage requirements and the method SOPs for additional storage information. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

## **22.4      DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS**

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to SOP No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in the analytical laboratory. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to SOP KNOX-QA-0001, Standard/Reagent Labeling and Documentation.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96%, a correction will be made to concentrations applied to solutions prepared from the stock commercial material. (For 1613B dioxin/furan analyses the purity must be 98% or corrections must be made.)

**22.4.1**      All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the analytical group standard tracking system. The following information is typically recorded in the group standard receipt logbook or in the certificate file.

- Standard/Reagent/Chemical Name
- Lot Number (Standard Identification Number)
- Vendor/Manufacturer
- Receipt Date
- Expiration Date
- Date Opened (only needed if expiration date is based on date opened)
- Recommended Storage Conditions (if not indicated on certificate or SOP)

Note: All solvents and acids may be stored at ambient temperature unless otherwise specified in the SOPs.

Records are maintained in standards logbooks for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs and as described below.

**22.4.2**      The preparation of standards and reagents is documented in standards and prepared reagents logbooks. The following information is documented:

- Unique, Traceable Standard or Reagent ID
- Preparation Date
- Preparer's Initials
- Compound/Element/Chemical/Standard Name or Description
- Manufacturer/Lot Number
- Expiration Date
- Concentration of stock standard (initial concentration)
- Volume/Weight (of stock standard used)
- Final Volume
- Final Concentration
- Solvent/Acid Used and the Lot Number (not needed if aqueous)

Note: The last five items may serve as a reference to the method of preparation.

**22.4.3** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date
- Standard ID
- Special Health/Safety warnings if applicable (Refer to the Health and Safety Manual)

Note: Refer to SOP KNOX-QA-0001 for more details.

**22.4.4** In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods and 3) according to the analytical SOPs.

## **SECTION 23.0**

### **SAMPLING (NELAC 5.5.7)**

#### **23.1 OVERVIEW**

TestAmerica Knoxville does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory. TestAmerica Knoxville also provides pre-cleaned sampling media for sample collection (e.g. source emission testing air sampling media).

#### **23.2 SAMPLING CONTAINERS**

The laboratory offers clean sampling containers for use by clients. The laboratory purchases clean containers from ESS and I-Chem (ESS QC class and I-Chem 300 Series containers). Certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

##### **23.2.1 Preservatives**

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade (for Volatiles with TerraCore)
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent (for Volatiles with TerraCore)
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent

##### **23.2.2 Preparing Container Orders**

Upon request, appropriate containers are sent to clients for use in collecting samples. When a client requests containers, a project manager/client services representative creates a bottle order form. The form is completed by the associate preparing the bottle order. The shipping date, type, number and lot of containers and preservatives are documented on the bottle order form. The original goes to the client with the containers; one copy is filed in the shipping area. Refer to SOP KNOX-SC-0006 for more details on bottle order preparation and shipping requirements

The laboratory also provides EnCore, TerraCore or other soil sampling devices when requested.

If containers are provided directly to the client from the manufacturer or from other sources, the laboratory will not be responsible for any of the above records.

### **23.3      FIELD QUALITY CONTROL (QC)**

Common field quality control samples are defined in the following paragraphs. The frequency of field quality control samples should be specified in the site specific Quality Assurance Project Plan (QAPP) or by the client. TestAmerica provides trip blanks for VOC analysis with the sample containers for all volatile organic analyses. Blanks generated in the field will be analyzed along with the field samples (exception soil samples where the blank is aqueous).

**23.3.1      Equipment Blank / Rinsate Blank** - The equipment blank, sometimes referred to as a rinsate blank, is a sample of the water used to decontaminate sampling equipment. The source water should be as free of target analytes as possible. An aliquot of this water is poured over or through the sample collection device after decontamination, collected in a sample container, preserved with appropriate reagents, and returned to the laboratory. This serves as a check on sampling device cleanliness, and will also be affected by the site and sample handling conditions evaluated by the other types of blanks. The sampling time for the equipment blank should begin when the equipment is rinsed and the water is collected.

**23.3.2      Field Blank** - The field blank is water that is as free of target analytes as possible and from the same source as the equipment blank. The water is poured into a sampling container at the sampling site, preserved with the appropriate reagents, and returned to the laboratory. This serves as a check on reagent and environmental contamination. The sampling time for the field blank should be when the blank is prepared in the field.

**23.3.3      Trip Blank** - The trip blank pertains to volatile analysis only. This serves as a check on sample contamination originating from sample transport, sample container contamination, shipping and storage, or from certain site conditions. Trip blanks are often referred to as travel blanks. They are prepared using pre-cleaned sample containers. They are filled with organic-free water (the source of the organic free water is the same source of water used to prepare volatile standards, method blanks, LCS and sample dilutions), sealed and taken into the field with the empty containers which will be used for sampling. The recommended frequency is one trip blank per cooler (in duplicate or triplicate), per volatiles method. Unless otherwise specified, the sampling time for the trip blank is the time of receipt at the laboratory (When the "Trip" ends).

**23.3.4      Field Duplicates** - Field duplicates are replicate samples collected from the same sampling point or location during a field collection event. This control sample is used to demonstrate the ability of both the sampling and analytical process to generate data of acceptable precision.

### **23.4      DEFINITION OF HOLDING TIME**

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis.



**23.4.1**     Semi-Volatile - Holding times for sample preparation for semi-volatile organics are measured from the sampling date until the day solvent contacts the sample. Holding times for analysis are measured from the date of initiation of extraction to the date of injection into the gas chromatograph.

**23.4.2**     Volatiles - Holding times for volatile organics are measured from the date (and time where applicable) of sampling to the date of injection into the gas chromatograph. Holding times for Encore preservation of volatiles samples is measured from the date and time of sampling to the date and time of sample preservation.

**23.4.3**     Inorganics - For inorganic and metals analysis, the preparation/digestion/distillation must be started within the maximum holding time as measured from the sampling date (and time where applicable). TestAmerica Knoxville measures the holding time for inorganics from sampling date (and time where applicable) to analysis date (and time where applicable).

## **23.5**        **SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES**

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times (refer to Tables 23-1 to 23-6) -or- preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

## **23.6**        **SAMPLE ALIQUOTS / SUBSAMPLING**

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis. In that regard the following guidelines apply to analysts:

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Refer to SOP KNOX-QA-0008, Subsampling, for details on the procedures to obtain representative subsamples for analysis from the samples submitted by the client.

Tables 23-1 to 23-6 detail holding times, preservation and container requirements, and sample volumes for SDWA and NPDES methods. The sample volumes are intended to be a minimal amount to perform the method, the containers that are used may be of larger size. **Please Note:** *the holding times are program specific and different programs may have different holding times for equivalent methods (e.g., there are difference in Holding times for many Organic analytes between SDWA and NPDES. RCRA methods may also be different).*

**Table 23-1.**  
**Holding Times, Preservation and Container Requirements: Drinking Water (SDWA)**

PARAMETER	CONTAINER	PRESERVATION <sup>1,2</sup>		HOLDING TIME <sup>3</sup>	SAMPLE VOLUME
		Temp. <sup>9</sup>	Chemical		
Cyanide	Plastic/Glass	4°C	NaOH to pH >12 Ascorbic acid <sup>8</sup> or Sodium arsenite <sup>8</sup>	14 days	500 mL
Fluoride	Plastic/Glass	None	None	None	250 mL
Mercury	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	28 days	250 mL
Metals <sup>4</sup>	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	6 months	250 mL
Nitrate	Plastic/Glass	4°C	None	48 hours <sup>5</sup>	250 mL
Nitrate-Nitrite <sup>6</sup>	Plastic/Glass	None	H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	250 mL
Nitrite	Plastic/Glass	4°C	None	48 hours	250 mL
Dioxin	Glass <sup>7</sup>	4°C	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	1 year	1 L

**Key to Table**

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater; and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
4. All metals except Hg.
5. If the sample is chlorinated, the holding time for an un-acidified sample kept at 4°C is extended to 14 days.
6. Nitrate-Nitrite refers to a measurement of total nitrite.
7. With Teflon lined septum.
8. If chlorinated add reagent prior to acidification (for Cyanide, add before NaOH).
9. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to < 6°C is acceptable.

**Table 23-2**  
**Holding Times, Preservation and Container Requirements: NPDES - Inorganic**

PARAMETER	CONTAINER <sup>1</sup>	PRESERVATION <sup>2,3</sup>		HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
		Temp <sup>13</sup>	Chemical		
Boron	Plastic <sup>5</sup>	None	HNO <sub>3</sub> to pH<2	6 months	200 mL
Bromide	Plastic/Glass	None	None	28 days	100 mL
Chloride	Plastic/Glass	None	None	28 days	50 mL
Cyanide – <u>Total</u> <sup>14,15</sup>	Plastic/Glass	≤ 6°C	NaOH to pH >12, 0.6 g ascorbic Acid <sup>7</sup>	14 days	100 mL
Fluoride	Plastic	None	None	28 days	300 mL
Hardness	Plastic/Glass	None	HNO <sub>3</sub> to pH<2 <sup>8</sup>	6 months	100 mL
Hydrogen Ion (pH)	Plastic/Glass	None	None	15 min. <sup>6</sup>	200 mL
Mercury <sup>11</sup>	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	28 days	200 mL
Metals <sup>9,10</sup>	Plastic/Glass	None	HNO <sub>3</sub> to pH<2 <sup>16</sup>	6 months	200 mL
Nitrate	Plastic/Glass	≤ 6°C	None	48 hours	100 mL
Nitrate-Nitrite	Plastic/Glass	≤ 6°C	H <sub>2</sub> SO <sub>4</sub> to pH <2	28 days	100 mL
Nitrite	Plastic/Glass	≤ 6°C	None	48 hours	100 mL
Orthophosphate	Plastic/Glass	≤ 6°C	Filter within 15 min.	48 hours	250 mL
Sulfate	Plastic/Glass	≤ 6°C	None	28 days	250 mL

**Key to Table**

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at ≤ 6°C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater; and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
5. May also be collected in quartz or PTFE Plastic.
6. For compliance testing, the analysis must be performed in the field at the time of analysis. If transported to the laboratory for analysis, the analysis will be performed as soon as practical and reported qualified.

**Key to Table**

7. Should only be used in the presence of residual chlorine. (Alternatively, sodium arsenite may be used)
8. H<sub>2</sub>SO<sub>4</sub> to a pH <2 is also acceptable.
9. Except Mercury and Hexavalent Chromium.
10. For dissolved metals, samples must be filtered on site before adding HNO<sub>3</sub> preservative (or before shipping to laboratory).
11. Samples collected for determination of trace level mercury (100 ng/L) using EPA 1631 must be collected in tightly capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipping, samples should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. Samples that been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.
13. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of “≤ °C” is used in place of the “4 °C” and “<4 °C” sample temperature requirements listed in some methods.
14. In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH. If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered (with sulfide treatment by laboratory) and qualify the results in the final report.
15. It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.
16. Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.

**Table 23-3**  
**Holding Times, Preservation and Container Requirements: NPDES - Organic**

PARAMETER	CONTAINER	PRESERVATION <sup>1,2</sup>		HOLDING TIME <sup>3</sup>	SAMPLE VOLUME
		Temp. <sup>7</sup>	Chemical		
CDD/CDFs <sup>6</sup> – Aqueous: Field/Lab Preservation	Glass	≤ 6°C	pH <9, 0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	1 year	1 L
CDD/CDFs <sup>6</sup> – Solids/Mixed Phase/ - Field Preservation	Glass	≤ 6°C	None	7 days <sup>8</sup>	1 L
CDD/CDFs <sup>6</sup> – Tissue – Field Preservation	Glass	≤ 6°C	None	24 hours <sup>8</sup>	10 grams
CDD/CDFs <sup>6</sup> – Solids/Mixed Phase/Tissue - Lab Preservation	Glass	< -10°C	None	1 year	1 L

**Key to Table**

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at ≤ 6°C until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater; and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
4. With Teflon lined septum.
5. Should only be used in the presence of residual chlorine. Ascorbic may be used instead.
6. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more categories, the sample may be preserved by cooling to ≤ 6°C reducing residual chlorine with 0.0008 % sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9. Samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine) and footnotes 10 and 11(re the analysis of Benzidine).
7. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of “≤ °C” is used in place of the “4 °C” and “<4 °C” sample temperature requirements listed in some methods.
8. Holding time from sampling field preservation at <6°C to time of preservation at <-10°C.

**Table 23-4.**  
**Holding Times, Preservation and Container Requirements: RCRA - Aqueous**

PARAMETER	CONTAINER <sup>1</sup>	PRESERVATION <sup>2,3</sup>		HOLDING TIME <sup>4</sup>	SAMPLE VOLUME
		Temp. <sup>12</sup>	Chemical		
Chloride	Plastic/Glass	4°C	None	28 days	100 mL
Cyanide -Total	Plastic/Glass	4°C	NaOH to pH >12 <sup>5</sup>	14 days	250 mL
Hydrogen Ion (pH)	Plastic/Glass	4°C	None	24 hours <sup>11</sup>	100 mL
Nitrate	Plastic/Glass	4°C	None	48 hours	28 days
Sulfate	Plastic/Glass	4°C	None	28 days	400 mL
Mercury	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	28 days	250 mL
Other Metals	Plastic/Glass	None	HNO <sub>3</sub> to pH<2	6 months	250 mL
Acrolein and Acrylonitrile	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> Adjust pH to 4-5 <sup>13</sup>	14 days	1 L
Benzidines	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L
Chlorinated Hydrocarbons	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L
Dioxins and Furans	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	30 days <sup>8</sup>	1 L
Haloethers	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L
Nitroaromatics and cyclic ketones	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> , store in dark	7 days <sup>8</sup>	1 L
Nitrosamines	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> , store in dark	7 days <sup>8</sup>	1 L
Organochlorine Pesticides	Glass <sup>10</sup>	4°C	None	7 days <sup>8</sup>	1 L
PCBs	Glass <sup>10</sup>	4°C	None	7 days <sup>8</sup>	1 L
Phenols	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	7 days <sup>8</sup>	1 L
Phthalate Esters	Glass <sup>10</sup>	4°C	None	7 days <sup>8</sup>	1 L
Polynuclear Aromatic Hydrocarbons	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> , store in dark	7 days <sup>8</sup>	1 L
Purgeable Hydrocarbons	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup> Adjust pH <2 <sup>2</sup>	14 days	40 mL
Purgeable Halocarbons	Glass <sup>10</sup>	4°C	0.0008 % Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>7</sup>	14 days	40 mL

**Key to Table**

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater; and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
5. If oxidizing agents are present, add 5 mL 0.1 N NaAsO<sub>2</sub> or 0.06 g of ascorbic acid per L. See Cyanide SOP for additional information about other interferences.
6. Adjust pH to <2 with H<sub>2</sub>SO<sub>4</sub>, HCl, or solid NaHSO<sub>4</sub>. Free Chlorine must be removed prior to adjustment.
7. Free Chlorine must be removed by the appropriate addition of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.
8. 7 days to extraction. 40 days from extraction to analysis. For Dioxins/Furans: 30 days to extraction 45 days from extraction to analysis.
9. Adjust pH to 5-8 using NaOH or H<sub>2</sub>SO<sub>4</sub>.
10. With Teflon lined septum.
11. Holding Time is listed as "As Soon as Possible" in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
12. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to ≤ 6°C is acceptable.
13. Based on guidance from EPA MICE, if samples are received without pH adjustment, the holding time is 7 days.



**Table 23-5.**  
**Holding Times, Preservation and Container Requirements: RCRA – Non-Aqueous**

PARAMETER	CONTAINER <sup>1</sup>	PRESERVATION Temp. <sup>7</sup>	Chemical	HOLDING TIME <sup>2</sup>	SAMPLE WEIGHT
Chloride	Glass	4°C	None	28 days	50 g
Cyanide -Total	Glass	4°C	None	14 days	50 g
Hydrogen Ion (pH)	Glass	4°C	None	7 days <sup>6</sup>	50 g
Nitrate	Glass	4°C	None	N/A	50 g
Oil and Grease	Glass	4°C	None	28 days	50 g
Sulfide	Glass	4°C	Add Zn Acetate, zero headspace	7 days	50 g
Chromium VI	Glass	4°C	None	30 days	50 g
Mercury	Plastic/Glass	None	None	28 days	50 g
Other Metals	Plastic/Glass	None	None	6 months	50 g
Acrolein and Acrylonitrile	Glass <sup>4</sup>	4°C	None	14 days	50 g
Benzidines	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Chlorinated Hydrocarbons	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Dioxins and Furans	Glass <sup>4</sup>	4°C	None	30 days <sup>3</sup>	50 g
Haloethers	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Nitroaromatics and cyclic ketones	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Nitrosamines	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Organochlorine Pesticides	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
PCBs	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Phenols	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Phthalate Esters	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Polynuclear Aromatic Hydrocarbons	Glass <sup>4</sup>	4°C	None	14 days <sup>3</sup>	50 g
Purgeable Hydrocarbons	Glass <sup>4</sup>	4°C	None	14 days <sup>5</sup>	50 g
Purgeable Halocarbons	Glass <sup>4</sup>	4°C	None	14 days <sup>5</sup>	50 g



**Key to Table**

1. Plastic should be Polyethylene.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
3. 14 days to extraction. 40 days from extraction to analysis. For Dioxins/Furans (Method 8290) in Matrices other than tissue: 30 days to extraction 45 days from extraction to analysis. For Dioxins/Furans (Method 8290) in Tissue: 30 days to extraction 45 days from collection to analysis
4. With Teflon Lined Septum
5. See Volatile SOP for more detailed preservation requirements.
6. Holding Time is listed as "As Soon as Possible" in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
7. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to  $\leq 6^{\circ}\text{C}$  is acceptable.

**Table 23-6.**  
**Holding Times, Preservation and Container Requirements: Air Samples<sup>1</sup>**

PARAMETER	CONTAINER	PRESERVATION		HOLDING TIME <sup>2</sup>	SAMPLE WEIGHT
		Temp.	Chemical		
Volatile Organics (TO-15, TO-14A)	Summa Canister	None	None	30 days	6L or 1L
Volatile Organics (TO-15, TO-14A)	Tedlar Bag	None	None	72 hrs <sup>3,4</sup>	1 L
Polynuclear Aromatic Hydrocarbons (TO-13A)	PUF/XAD	4°C <sup>5</sup>	None	7 days <sup>6</sup>	N/A
Organochlorine Pesticides/PCBs (TO-4A)	PUF/XAD	4°C <sup>5</sup>	None	7 days <sup>6</sup>	N/A
Dioxins/Furans (TO-9A)	PUF/XAD	4°C <sup>5</sup>	None	7 days <sup>6</sup>	N/A

**Key to Table**

1. For details on Source Air Emission Holding Times, Containers, and Preservation please contact the laboratory for method specific information.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
3. Holding Time is based on SW 846 Method 0040 "SAMPLING OF PRINCIPAL ORGANIC HAZARDOUS CONSTITUENTS FROM COMBUSTION SOURCES USING TEDLAR® BAGS". Some states specifically enforce this holding time (e.g. Florida, New Jersey) and others have not specified this information in their regulatory requirements.
4. The holding time is 72 hours unless the laboratory has a documented validation study that indicates a longer HT is acceptable for the analytes of interest. Alternatively, the sample may be transferred into a Summa Canister within 72 hours.
5. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to ≤ 6°C is acceptable.
6. 7 days from collection to extraction, 40 days from extraction to analysis.

## **SECTION 24**

### **HANDLING OF SAMPLES (NELAC 5.5.8)**

Sample management procedures at TestAmerica Knoxville ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

#### **24.1      CHAIN OF CUSTODY (COC)**

The COC form is the written documented history of any sample and is initiated at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 24-1.

##### **24.1.1      Field Documentation**

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 24-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

**Note:** Independent couriers are not required to sign the COC form.

#### **24.1.2     Legal / Evidentiary Chain-of-Custody**

TestAmerica Knoxville provides internal sample chain-of-custody tracking. However, it does not provide chain-of-custody evidence for samples identified for legal/evidentiary purposes as defined by the 2003 NELAC Standard Section 5.4.12.

### **24.2     SAMPLE RECEIPT**

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

#### **24.2.1     Laboratory Receipt**

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Condition Upon Receipt Variance Form (See Example form in Figure 24-3). Details on the receipt of samples are found in SOP KNOX-SC-0003, Sample Receipt and Log-in.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

#### **24.2.2     Sample Log-in**

All samples that are received by the laboratory are logged into the LIMS to allow the laboratory to track and evaluate sample progress. Each group of samples that are logged in together (typically one project from a given client/sampling event) is assigned a unique Lot number. Within each Lot, each sample receives a unique Work Order number. A sample may be composed of more than one bottle since different preservatives may be required to perform all analyses requested. If multiple containers are received for a single sample, each container is uniquely identified with a numerical container number added to the sample number. The LIMS generates sample labels that are attached to each bottle for a given sample.

Each lot of samples is logged into LIMS with the following information:

- Client Name, Project Name, Address, Phone, Fax (where necessary), Report to information, invoice to information (most of this information is “default information” that is stored in the LIMS);
- Date and time sampled;
- Date and time received;
- Job and/or project description, sample description;
- Sample matrix, special sample remarks;
- Reporting requirements (i.e., QC level, report format, invoicing format);
- Turn-around-time requirements;
- Parameters (methods and reporting limits or MDLs are default information for a given parameter).

Some of this information is set up in the LIMS quote by the project manager before sample log-in occurs. See SOP KNOX-SC-0003, Sample Receipt and Log-in for further details on sample log-in requirements.

### **24.3 SAMPLE ACCEPTANCE POLICY**

The laboratory has a written sample acceptance policy (Figure 24-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method;
- sample holding times must be adhered to;
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- the client will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

### **24.4 SAMPLE STORAGE**

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators or freezers suitable for the sample matrix. Samples for methods that do not require refrigeration (e.g., aqueous samples for ICP metals analysis) may be stored on shelves at ambient temperature. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts retrieve the sample container(s) allocated to their analysis from the designated storage location, analyze the sample, and return the remaining sample to the storage location from which it originally came. Residual samples are maintained in the designated storage location until disposal at least thirty days after the report is issued. (Exception: TO-14A or TO-15 canister samples are subject to disposal two days after the report is issued.) Special arrangements may be made to store samples for longer periods of time.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

#### **24.5      HAZARDOUS SAMPLES AND FOREIGN SOILS**

All samples are assumed to be hazardous. All samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm. All soil samples are sent out for incineration by a USDA-approved waste disposal facility.

#### **24.6      SAMPLE SHIPPING**

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

#### **24.7      SAMPLE DISPOSAL**

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: KNOX-SC-0005, Sample Disposal). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

### Figure 24-1. Example: Chain of Custody (COC)

Knoxville, TN 37921

## Chain of Custody Record



phone 865-291-3000

TestAmerica Laboratories, Inc.

[illegible]

**Figure 24-2. Example: Sample Acceptance Policy**  
**TestAmerica Knoxville**  
**Sample Acceptance Policy**

NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. TestAmerica Knoxville will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

When completing the chain of custody form, please do not forget to sign your name in the "relinquished by" box.

NELAC requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided. (Chain of Custody filled out properly)
- Each sample shall be labeled with unique, durable and indelible identification, (See note below regarding SUMMA canisters and VOST tubes).
- The samples shall be collected in the appropriate sample containers.
- The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses and associated quality control.
- All samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time
- The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation.

NOTE: Never affix a label directly on a SUMMA canister. A special tag is attached to each canister for this purpose. Never place a label on a VOST tube; use the external container for labeling.

NOTE: Canisters and flow controllers must be returned even if they were not used. Sampling equipment that is lost or not returned will be invoiced to the client at the replacement costs.

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Figure 24-3. Example: Condition Upon Receipt Form

Client: _____		Project: _____		Lot Number: _____	
Review Items	Yes	No	NA	If No, what was the problem?	Comments/Actions Taken
1. Do sample container labels match COC? (IDs, Dates, Times)				<input type="checkbox"/> 1a Do not match COC <input type="checkbox"/> 1b Incomplete information <input type="checkbox"/> 1c Marking smeared <input type="checkbox"/> 1d Label torn <input type="checkbox"/> 1e No label <input type="checkbox"/> 1f COC not received <input type="checkbox"/> 1g Other: _____	
2. Is the cooler temperature within limits? (> freezing temp. of water to 6 °C; NC, 1668, 1613B: 0-4 °C; VOST: 10 °C; MA: 2-6 °C)				<input type="checkbox"/> 2a Temp Blank = _____ <input type="checkbox"/> 2b Cooler Temp = _____	
3. Were samples received with correct chemical preservative (excluding Encore)?				<input type="checkbox"/> 3a Sample preservative = _____	
4. Were custody seals present/intact on cooler and/or containers?				<input type="checkbox"/> 4a Not present <input type="checkbox"/> 4b Not intact <input type="checkbox"/> 4c Other: _____	
5. Were all of the samples listed on the COC received?				<input type="checkbox"/> 5a Samples received-not on COC <input type="checkbox"/> 5b Samples not received-on COC	
6. Were all of the sample containers received intact?				<input type="checkbox"/> 6a Leaking <input type="checkbox"/> 6b Broken	
7. Were VOA samples received without headspace?				<input type="checkbox"/> 7a Headspace (VOA only)	
8. Were samples received in appropriate containers?				<input type="checkbox"/> 8a Improper container	
9. Did you check for residual chlorine, if necessary?				<input type="checkbox"/> 9a Could not be determined due to matrix interference	
10. Were samples received within holding time?				<input type="checkbox"/> 10a Holding time expired	
11. For rad samples, was sample activity info. provided?				<input type="checkbox"/> Incomplete information	
12. For SOG water samples (1613B, 1668A, 8290, LR PAHs), do samples have visible solids present?				If yes & appears to be > 1%, was SOG notified? _____	
13. Are the shipping containers intact?				<input type="checkbox"/> 13a Leaking <input type="checkbox"/> 13b Other: _____	
14. Was COC relinquished? (Signed/Dated/Timed)				<input type="checkbox"/> 14a Not relinquished	
15. Are tests/parameters listed for each sample?				<input type="checkbox"/> 15a Incomplete information	
16. Is the matrix of the samples noted?				<input type="checkbox"/> 15a Incomplete information	
17. Is the date/time of sample collection noted?				<input type="checkbox"/> 15a Incomplete information	
18. Is the client and project name/# identified?				<input type="checkbox"/> 15a Incomplete information	
19. Was the sampler identified on the COC?				<input type="checkbox"/> 15a Incomplete information	
Quote #: _____ PM Instructions: _____					
Sample Receiving Associate: _____ Date: _____					

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## SECTION 25.0

### ASSURING THE QUALITY OF TEST RESULTS (NELAC 5.5.9)

#### 25.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 21, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

#### 25.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, solvent extraction, sonication, acid digestion, distillation, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discrete manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

#### 25.3 NEGATIVE CONTROLS

**25.3.1 Method Blanks** are used to assess preparation and analysis for possible contamination during the preparation and processing steps.

**25.3.1.1** The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.

**25.3.1.2** The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).

**25.3.1.3** The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.

**25.3.1.4** Evaluation criteria and corrective action for method blanks is defined in the specific standard operating procedure for each analysis. Generally, corrective action is taken if the concentration of a target analyte in the blank is at or above the reporting limit as established in the SOP for the method or regulation. For work done in support of the DOD QSM, one half the reporting limit is used as the acceptance criteria for the method blank (<RL for common laboratory contaminants).

- The source of contamination is investigated.
- Measures are taken to minimize or eliminate the source of the contamination.
- Affected samples are reprocessed or the results are qualified on the final report.

**25.3.2** **Calibration Blanks** are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

**25.3.3** **Instrument Blanks** are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

**25.3.4** **Trip Blanks** are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. A trip blank is prepared by the laboratory by filling a clean container with pure deionized water that has been purged or filtered to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples. Trip Blanks are also sometimes referred to as Travel Blanks.

**25.3.5** **Field Blanks** are sometimes used for specific projects by the field samplers. A field blank is prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

**25.3.6** **Equipment Blanks** are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

**25.3.7** **Holding Blanks**, also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory (refer to Section 24.4).

**25.3.8** Field blanks, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. When known, blanks should not be selected for matrix QC, as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB".

## **25.4** **POSITIVE CONTROLS**

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike

(MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch.

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are listed in each analytical SOP.

#### **25.4.1 Method Performance Control - Laboratory Control Sample (LCS)**

**25.4.1.1** The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

**25.4.1.2** The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, and taken through all preparation and analysis steps along with the field samples. Where there is no preparation for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances (e.g., ICP metals in soil), aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids.

**25.4.1.3** As stated in the opening of this section, the LCS goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).

**25.4.1.4** The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

**25.4.1.5** If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g., no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

**25.4.1.5.1** For methods that have 1-10 target analytes, spike all components.

**25.4.1.5.2** For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

**25.4.1.5.3** For methods with more than 20 target analytes, spike at least 16 components.

**25.4.1.5.4** Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

**25.4.1.5.5** Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project specific basis.

**25.4.1.6** **Accuracy Calculation:** Percent Recovery (%R) Calculation (applies to LCS, CCV, Surrogates, and Matrix Spikes).

$$\% R = \frac{AV}{TV} \times 100$$

Where: AV = Analyzed Value  
TV = True Value

## **25.5** **SAMPLE MATRIX CONTROLS**

### **25.5.1** **Matrix Spikes (MS)**

**25.5.1.1** The Matrix spike is used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used.

**25.5.1.2** An MS is essentially a sample fortified with a known amount of the test analyte(s). At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.

**25.5.1.3** If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number of the listed components (see LCS analytes 25.4.1.5 above) may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit-specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

**25.5.1.4** The percent recovery calculation for matrix spikes is essentially the same as the calculation shown in 25.4.1.6 except that:

$$AV = Sp - Sa$$

Where: Sp = Spike result  
Sa = Sample result

## **25.5.2 Surrogate Spikes**

**25.5.2.1** Surrogate Spikes are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.

**25.5.2.2** Surrogate compounds are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method (also refer to Section 25.6.8). Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery. Repreparation and reanalysis of the client samples with failed surrogate recovery is performed if there is sufficient sample and the holding time has not expired. If the sample holding time has expired, the client should be consulted prior to repreparation and reanalysis.

## **25.5.3 Duplicates**

**25.5.3.1** For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure. Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

### **25.5.3.2 Precision Calculation (Relative Percent Difference - RPD)**

$$RPD = \frac{|S - D|}{\frac{(S + D)}{2}} \times 100$$

Where: S=Sample Concentration  
D=Duplicate Concentration

## **25.5.4 Internal Standards**

**25.5.4.1** In most organic analyses, internal standards are spiked into all environmental and quality control samples (including the initial calibration standards). An internal

standard is also used with some metals analyses. It is added to sample extracts after the extraction (post-prep). The acceptance criteria in most methods are 50% to 200% of the responses in the mid-point of the corresponding calibration curve. Consult the method-specific SOPs for details on the internal standard compounds, calculations and acceptance criteria. (Note: Internal standards are added prior to sample extraction for isotope dilution test methods.)

- 25.5.4.2** When the internal standard recoveries fall outside these limits, if there are not obvious chromatographic interferences, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets internal standard recovery criteria, the second run is reported (or both are reported if requested by the client). Refer to the analytical SOPs for internal standard criteria and corrective actions for each method.

## **25.6      ACCEPTANCE CRITERIA (CONTROL LIMITS)**

**25.6.1** Each individual analyte in the LCS, MS, or Surrogate Spike are evaluated against the control limits as published in the test method. Where there are no established acceptance criteria, the laboratory calculates control limits with the use of control charts or, in some cases, utilizes client project specific or regulatory mandated control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

**Note:** For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

**25.6.2** Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating (e.g. EPA SW846 8000 series methods where required by client or program). Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

**25.6.2.1** The lab should consider the effects of the spiking concentration on control limits, and to avoid censoring of data. The acceptance criteria for recovery and precision are often a function of the spike concentration used. Therefore, caution must be used when pooling data to generate control limits.

**25.6.2.2** Not only should the results all be from a similar matrix, but the spiking levels should also be approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results should all be generated using the same set of extraction, cleanup and analysis techniques. For example, results from solid samples extracted by ultrasonic extraction are not mixed with those extracted by Soxhlet.

**25.6.2.3** The laboratory must try and avoid discarding data that do not meet a preconceived notion of acceptable performance. This results in a censored data set, which, when used to develop acceptance criteria, will lead to unrealistically narrow criteria. For a 99% confidence interval, 1 out of every 100 observations likely will still fall outside the limits. For methods with long analyte lists, this may mean occasional failures



every batch or two. While professional judgment is important in evaluating data to be used to develop acceptance criteria, specific results are not discarded simply because they do not meet one's expectations. However, data points shall be discarded if they were the result of human or mechanical error or sample concentration exceeded spike level by  $> 4x$ .

**25.6.3** Laboratory generated % Recovery acceptance (control) limits are generally established by taking  $\pm 3$  Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

**25.6.3.1** Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV) limits (unless the analytical method specifies a tighter limit).

**25.6.3.2** In-house limits cannot be any wider than those mandated in a regulated analytical method (e.g., SDWA Dioxin Method 1613B 2,3,7,8-TCDD on-going precision and recovery limits of 73% to 146% recovery).

**25.6.4** The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

**25.6.5** Refer to SOP KNOX-QA-0004, Control Limits and Control Charting for details on the generation of control limits and the use of control charts at TestAmerica Knoxville.

**25.6.6** A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

**25.6.6.1** The analyte results are below the reporting limit and the LCS is above the upper control limit.

**25.6.6.2** If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

**25.6.6.3** For Department of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME). The use of allowable marginal exceedances may also be used for evaluation of LCS recoveries for NELAC accredited methods:

- $< 11$  analytes – 0 marginal exceedances are allowed.
- 11 – 30 Analytes – 1 marginal exceedance is allowed
- 31-50 Analytes – 2 marginal exceedances are allowed
- 51-70 Analytes – 3 marginal exceedances are allowed
- 71-90 Analytes – 4 marginal exceedances are allowed
- $> 90$  Analytes – 5 marginal exceedances are allowed



**25.6.6.3.1** Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC). (Note: The DOD QSM has defined limits for marginal exceedances. Refer to the DOD QSM SOP KNOX-QA-0021, DOD QSM Program Requirements.)

**25.6.6.3.2** Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random. Refer to the DOD QSM SOP KNOX-QA-0021, DOD QSM Program Requirements for details on how the laboratory ensures that the marginal exceedances are random.

**25.6.6.3.3** Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

**25.6.7** If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the analytical SOPs and in Section 13.

**25.6.8** If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client).

## **25.7      METHOD DETECTION LIMITS (MDLs)**

MDLs, calculated as described in Section 20.7, are updated or verified annually, or more often if required by the method.

## **25.8      ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL**

**25.8.1** The laboratory has written procedures to assure the accuracy of the test method including calibration (see Section 21), use of certified reference materials (see Section 22) and use of PT samples (see Section 16).

**25.8.2** A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 20.

**25.8.3** Use of formulae to reduce data is discussed in the method standard operating procedures and in Section 21.

**25.8.4** Selection of appropriate reagents and standards is included in Section 9 and 22.

**25.8.5** A discussion on selectivity of the test is included in Section 5.

**25.8.6** Constant and consistent test conditions are discussed in Section 19.

**25.8.7** The laboratories sample acceptance policy is included in Section 24.

## **SECTION 26.0**

### **REPORTING RESULTS (NELAC 5.5.10)**

#### **26.1      OVERVIEW**

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is a conflict between the client requested formats and accreditation requirements or data usability information, accreditation requirements and data usability information will take precedence over client requests. A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 20.

#### **26.2      TEST REPORTS**

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

**26.2.1**      A report title (e.g., Analytical Report) on the cover page with a "Result" column header on the sample results page.

**26.2.2**      Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

**26.2.3**      A unique identification of the report (e.g., Lot Number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:**      Page numbers of reports are represented on the upper right hand corner of each page. A table of contents precedes the cover page on each report which includes the page number of each section as well as the total number of pages.

**26.2.4** A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

**26.2.5** The name and address of client and a project name/number, if applicable.

**26.2.6** Client project manager or other contact.

**26.2.7** Description and unambiguous identification of the tested sample(s) including the client identification code.

**26.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

**26.2.9** Date reported or date of revision, if applicable.

**26.2.10** Method of analysis including method code (EPA, Standard Methods, etc).

**26.2.11** Reporting limit or Minimum Level.

**26.2.12** Method detection limits or Estimated Detection Limits (if requested).

**26.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).

**26.2.14** Sample results.

**26.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD (if applicable) recoveries and control limits.

**26.2.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets. This is discussed in the narrative and a copy of the condition upon receipt form is included with the chain of custody.

**26.2.17** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

**26.2.18** A statement that the report shall not be reproduced except in full, without the written approval of the laboratory.

**26.2.19** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

**26.2.20** When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

**26.2.21** Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

**26.2.22** When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

**26.2.23** Appropriate laboratory certification number for the state of origin of the sample, if applicable.

**26.2.24** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary report), and that a complete report will follow once all of the work has been completed.

**26.2.25** Any out of network subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All in-network subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

**26.2.26** Ohio EPA VAP requires that an affidavit must accompany each analytical report.

### **26.3 REPORTING LEVEL OR REPORT TYPE**

TestAmerica Knoxville offers four standard types of data report format.

- A Standard Report is a report containing the items in Section 26.2 above.
- A CLP Forms Only Report is a standard report using CLP (or CLP like) report forms.
- An Expanded Deliverable Report is a Standard Report which includes the supporting raw data.
- A CLP Expanded Deliverable Report is an Expanded Deliverable report using CLP (or CLP-like) report forms.

Further details, including other specific reporting options, are defined in SOP KNOX-AD-0004, Data Reporting.

#### **26.3.1 Electronic Data Deliverables (EDDs)**

EDDs are routinely offered as part of TestAmerica’s services. TestAmerica Knoxville offers a variety of EDD formats including industry standard and client specific electronic deliverables.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

### **26.4 SUPPLEMENTAL INFORMATION FOR TEST**

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a

narrative explaining the discrepancy in the front of the report. Refer to Appendix 7 for a list of the laboratory's standard footnotes and qualifiers.

**26.4.1** Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

**26.4.2** Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

**26.4.3** Where applicable, a statement on the estimated uncertainty of measurements is provided; information on uncertainty is needed when a client's instructions so require.

**26.4.4** Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

## **26.5      ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS**

If TestAmerica Knoxville is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of the TestAmerica network are reported to the client as provided from the subcontract laboratory.

## **26.6      CLIENT CONFIDENTIALITY**

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

**Note:** This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

**Note:** Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

**26.6.1** Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

Confidentiality Notice: The information contained in this message is intended only for the use of the addressee, and may be confidential and/or privileged. If the reader of this message is not the intended recipient, or the employee or agent responsible to deliver it to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify the sender immediately.

## **26.7      FORMAT OF REPORTS**

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

## **26.8 AMENDMENTS TO TEST REPORTS**

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 13).

The revised report is retained on the archive data server, as is the original report. The revised report is stored in the archive data server under the lot number followed by "rev". The revised report will have the word "revised" on the cover page.

When the report is re-issued, a notation of "Reissued Report" is placed on the cover/signature page of the report or at the top of the narrative page with a brief explanation of reason for the re-issue.

## **26.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS**

### **26.9.1 Sample Reanalysis Policy**

Because there is a certain level of uncertainty with any analytical measurement a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client specific arrangements for reanalysis protocols can be established.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within  $\pm 1$  reporting limit for samples  $\leq 5x$  the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report, but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Group Leader or Laboratory Director if unsure.

### **26.9.2 Policy on Data Omissions or Reporting Limit Increases**

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:



- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely no possible impact on the interpretation of the analytical results and there is no possibility of the change being interpreted as misrepresentation by anyone inside or outside of our company.

### **26.9.3     Multiple Reports**

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

## Appendix 1.

# TESTAMERICA ETHICS POLICY No. CA-L-P-001

Refer to CA-L-P-001 for complete policy.

## **TestAmerica** **EMPLOYEE ETHICS STATEMENT**

*I understand that TestAmerica is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.*

- With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:*
- I will not intentionally report data values that are inconsistent with the actual values observed or measured.*
- I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations.*
- I will not intentionally misrepresent another individual's work as my own or represent my own work as someone else's.*
- I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers; I shall not modify data (either sample or QC data) unless the modification can be technically justified through a measurable analytical process, such as one deemed acceptable to the laboratory's Standard Operating Procedures, Quality Assurance Manual or Technical Director. All such modifications must be clearly and thoroughly documented in the appropriate laboratory notebooks/worksheets and/or raw data and include my initials or signature and date.*
- I shall not make false statements to, or seek to otherwise deceive, members of Management or their representatives, agents, or clients/customers. I will not, through acts of commission, omission, erasure, or destruction, improperly report measurement standards, quality control data, test results or conclusions.*
- I shall not compare or disclose results for any Performance Testing (PT) sample, or other similar QA or QC requirements, with any employee of any other laboratory, including any other TestAmerica laboratory, prior to the required submission date of the results to the person, organization, or entity supplying the PT sample.*
- I shall immediately inform my supervisor or other member of management regarding any intentional or unintentional reporting of my own inauthentic data. Such report shall be given both orally and in writing to the supervisor or other member of management contacted and to the local Quality Assurance Manager. The Quality Assurance Manager will initial and date the information and return a copy to me. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and will immediately report its occurrence. If I have actual knowledge of such acts committed by any other employees, and I do not report such information to designated members of Management, it shall be considered as serious as if I personally committed the offense. Accordingly, in that event, I understand that I may be subject to immediate termination of employment.*
- I understand that if any supervisor, manager, or representative of TestAmerica management instructs, requests, or directs me to perform any of the aforementioned improper laboratory practices, or if I am in doubt or uncertain as to whether or not such laboratory practices are proper, I will not*

*comply. In fact, I must report such event to all appropriate members of Management including, but not limited to, the Lab Director, all supervisors and managers with direct line reporting relationship between me and the Lab Director, and the local Quality Assurance representative, excluding such individuals who participated in such perceived improper instruction, request, or directive. In addition, I may contact Corporate Quality Assurance / Ethics Compliance Officer(s) for assistance.*

- I understand the critical importance of accurately reporting data, measurements, and results, whether initially requested by a client, or retained by TestAmerica and submitted to a client at a later date, or retained by TestAmerica for subsequent internal use;*
- I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the TestAmerica family of companies.*
- I shall not accept gifts of a value that would adversely influence judgment.*
- I shall avoid conflicts of interest and report any potential conflicts to the management (e.g. employment or consulting with competitors, clients, or vendors).*
- I shall not participate in unfair competition practices (e.g. slandering competitors, collusion with other labs to restrict others from bidding on projects).*
- I shall not misrepresent certifications and status of certifications to clients or regulators.*
- I shall not intentionally discharge wastes illegally down the drain or onto the ground.*
- I understand that any attempt by management or an employee to circumvent these policies will be subject to disciplinary action.*

*As a TestAmerica employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.*

*I understand that if my job includes supervisory responsibilities, I shall not instruct, request, or direct any subordinate to perform any laboratory practice which is unethical or improper. Also, I shall not discourage, intimidate, or inhibit an employee who may choose to appropriately appeal my supervisory instruction, request, or directive which the employee perceives to be improper, nor retaliate against those who do.*

*The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination of my employment. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.*

EMPLOYEE SIGNATURE \_\_\_\_\_

Date \_\_\_\_\_

Supervisor/Trainer: \_\_\_\_\_

Date \_\_\_\_\_

Work Instruction No. CA-WI-005

**TestAmerica**  
**CONFIDENTIALITY AND PROPRIETARY INFORMATION AGREEMENT**

TestAmerica and their predecessors, in their businesses, have developed and use commercially valuable technical and non-technical information and to guard the legitimate interests of TestAmerica and its clients, it is necessary to protect certain information as confidential and proprietary.

I, \_\_\_\_\_, understand and acknowledge that during the term of my employment by TestAmerica, I will be privy to and entrusted with certain confidential information and trade secrets of TestAmerica and its clients.

*Confidential information and trade secrets include, but are not limited to: customer and client lists; price lists; marketing and sales strategies and procedures; operational and equipment techniques; standard operating procedures; business plans and systems; quality control procedures and systems; special projects and technological research, including projects, research and reports for any government entity or client; client's plans and processes; client's manner of operation; the trade secrets of clients; client's data; vendor or supplier pricing; employee lists and personal information, and any other records, data, files, drawings, inventions, discoveries, applications, or processes which are not in the public domain.*

I agree as follows:

1. I will not in any way, during the term of my employment, or at any time thereafter, except as authorized in writing by the Legal Department of TestAmerica or the client where client data is involved, disclose to others, use for my own benefit, remove from TestAmerica's premises (except to the extent off-site work is approved by my supervisor), copy or make notes of any confidential information and/or trade secrets of TestAmerica or its clients, excepting only that information which may be public knowledge. Technical and business information of any previous employer or other third party which I may disclose to TestAmerica shall be limited to that which was acquired legitimately and disclosed to me without restriction as to secrecy.
2. I agree that all inventions (whether or not patentable) conceived or made by me during the period of my employment by TestAmerica shall belong to TestAmerica, provided such inventions grow out of my work for TestAmerica and are related to the business of TestAmerica. I agree to disclose and assign such inventions to TestAmerica. In California, this provision shall not apply to any invention which qualifies fully under Section 2870 of the California Labor Code.
3. On termination of my employment from TestAmerica, I will deliver to TestAmerica all documents, records, notes, data, memoranda, files, manuals, equipment and things of any nature which relate in any way to confidential information and/or trade secrets of TestAmerica or its clients and which are in my possession or under my control.
4. I agree that during the period of my employment and for one (1) year from and after the termination (for any reason) of my employment with TestAmerica, I shall not directly or indirectly (without first obtaining the written permission of TestAmerica), recruit for employment, or induce to terminate his or her employment with TestAmerica, any person who is an active employee of TestAmerica on the last day of my employment with TestAmerica.
5. I acknowledge that if I were to breach any provision of this Confidentiality Agreement, money damages will be inadequate, and I hereby agree that TestAmerica shall be entitled, where appropriate, to specific performance and/or injunctive relief (i.e. to require me to comply with this Agreement). I further acknowledge that the willingness of TestAmerica to hire me or to continue my employment constitutes full and adequate consideration for the agreements, and obligations to which I have agreed as set forth in this document.

I have executed this Agreement, intending to be legally bound.

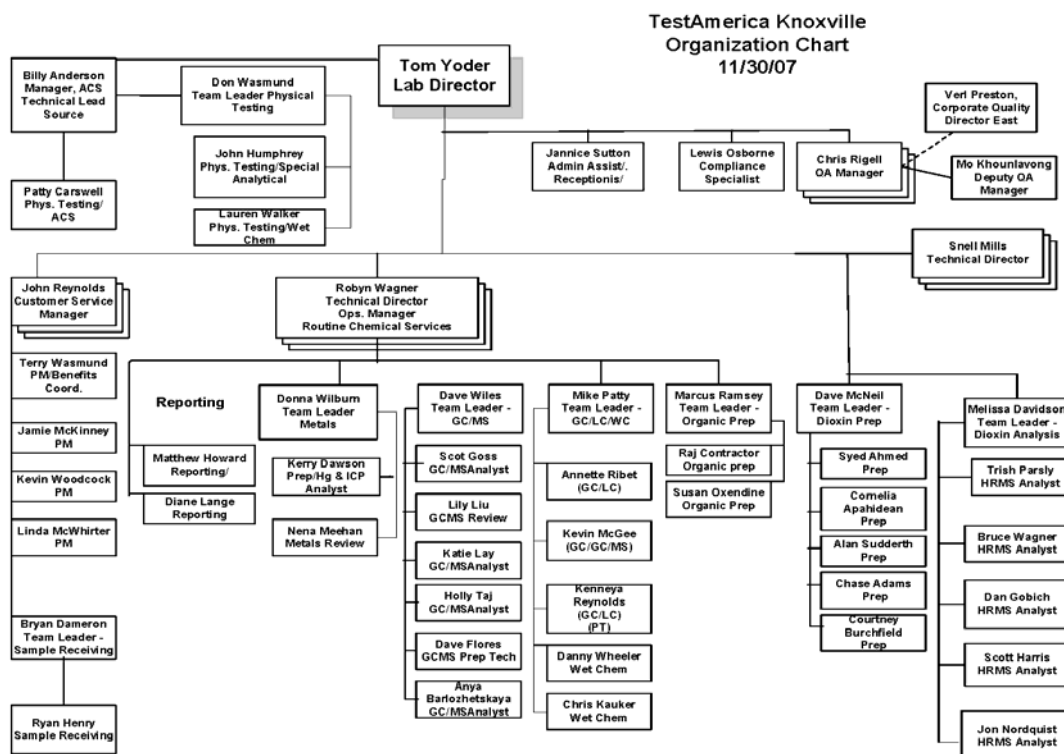
\_\_\_\_\_  
Printed Name

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date  
Work Instruction No. CA-WI-006

## Appendix 2.

### Example Laboratory Organization Chart

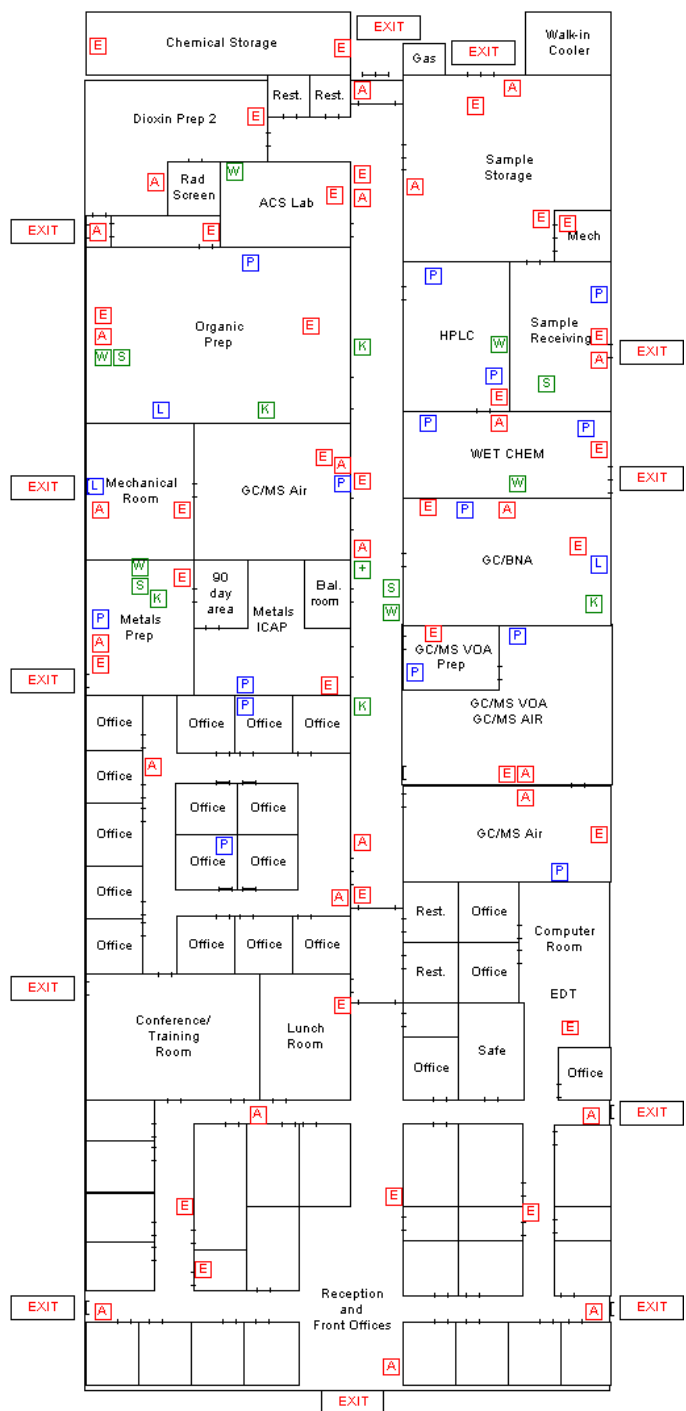


## Appendix 3. Laboratory Floor Plan

### TestAmerica Knoxville Laboratory Main Building - Figure 1

#### Safety Equipment Location

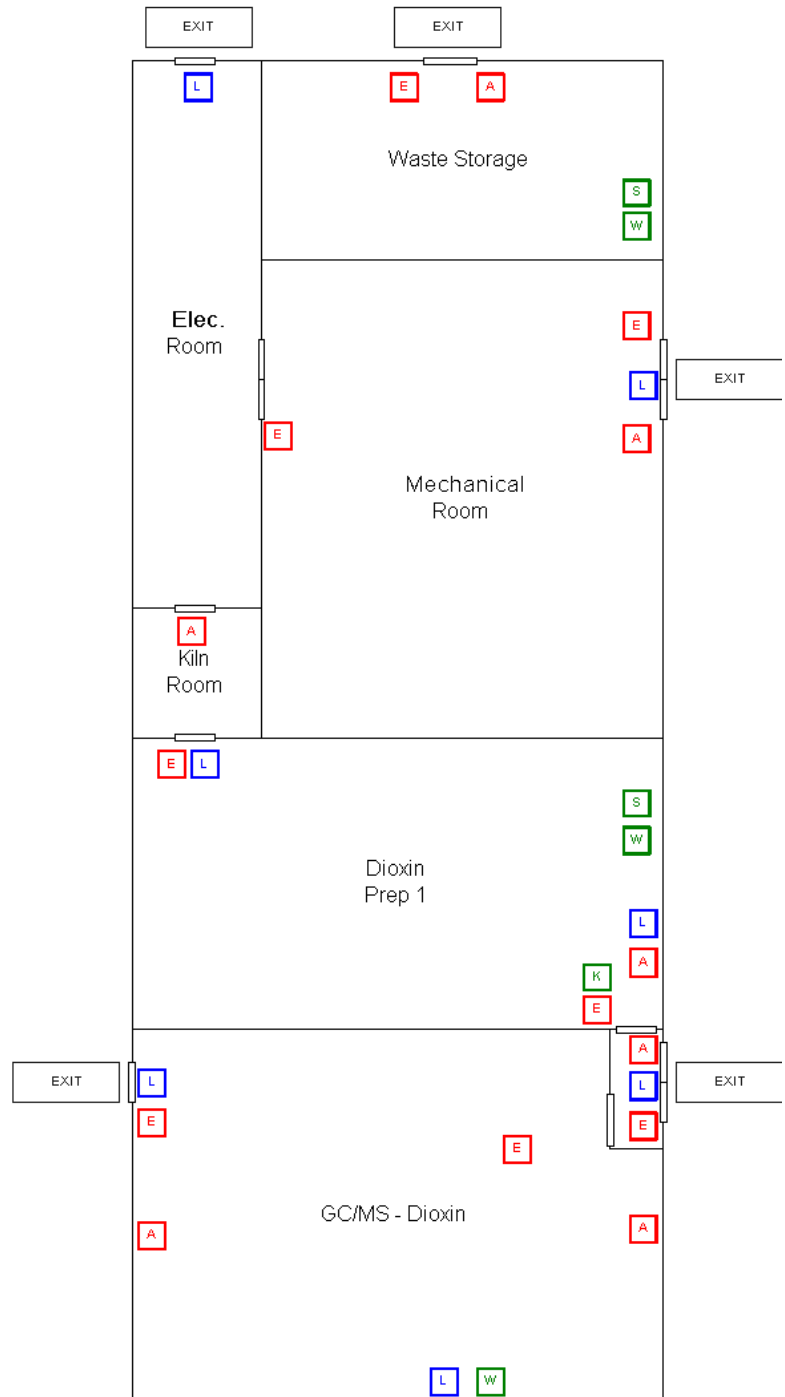
- E Fire Extinguisher
- A Fire Alarm or Siren
- + First Aid Kit
- S Safety Shower
- W Eye Wash Station
- K Emergency Spill Kit
- L Emergency Lighting - Wired
- P Emergency Lighting - Portable



**Appendix 3. Laboratory Floor Plan (continued)**  
**TestAmerica Knoxville Laboratory**  
**Building 2 - Dioxin Lab - Figure 2**

**Safety Equipment Location**

E	Fire Extinguisher
A	Fire Alarm or Siren
+	First Aid Kit
S	Safety Shower
W	Eye Wash Station
K	Emergency Spill Kit
L	Emergency Lighting - Wired
P	Emergency Lighting - Portable



Appendix 4. Reserved.



## **Appendix 5. Glossary/Acronyms**

### **Glossary:**

#### **Acceptance Criteria:**

Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

#### **Accreditation:**

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

#### **Accrediting Authority:**

The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

#### **Accuracy:**

The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

#### **Analyst:**

The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

#### **Assessment:**

The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

#### **Assessment Criteria:**

The measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

#### **Assessment Team:**

The group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (NELAC)

#### **Assessor:**

One who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (NELAC)

#### **Audit:**

A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

**Batch:**

Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

**Blank:**

A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

**Blind Sample:**

A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

**Calibration:**

To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

**Calibration Curve:**

The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

**Calibration Method:**

A defined technical procedure for performing a calibration. (NELAC)

**Calibration Standard:**

A substance or reference material used to calibrate an instrument (QAMS)

**Certified Reference Material (CRM):**

A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

**Chain of Custody:**

An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

**Clean Air Act:**

The enabling legislation in 42 U.S.C. 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

**Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):**

The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

**Compromised Samples:**

Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

**Confidential Business Information (CBI):**

Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

**Confirmation:**

Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

- Second column confirmation
- Alternate wavelength
- Derivatization
- Mass spectral interpretation
- Alternative detectors or
- Additional Cleanup procedures

(NELAC)

**Conformance:**

An affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

**Corrective Action:**

The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

**Data Audit:**

A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

**Data Reduction:**

The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

**Deficiency:**

An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

**Detection Limit:**

The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

**Document Control:**

The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

**Duplicate Analyses:**

The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

**Environmental Detection Limit (EDL):**

The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

**Equipment Blank:**

Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

**External Standard Calibration:**

Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

**Federal Insecticide, Fungicide and Rodenticide Act (FIFRA):**

The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (NELAC)

**Federal Water Pollution Control Act (Clean Water Act, CWA):**

The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

**Field Blank:**

Blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

**Field of Testing:**

NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

**Finding:**

An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (NELAC)

**Holding Times (Maximum Allowable Holding Times):**

The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

**Inspection:**

An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

**Internal Standard:**

A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

**Internal Standard Calibration:**

Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

**Instrument Blank:**

A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

**Instrument Response:**

Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

**Laboratory:**

A defined facility performing environmental analyses in a controlled and scientific manner. (NELAC)

**Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):**

A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in

aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:

Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Least Squares Regression (1<sup>st</sup> Order Curve):

The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD):

An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Manager (however named):

The individual designed as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

Matrix:

The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

**Biological Tissue:** any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

**Solids:** includes soils, sediments, sludges, and other matrices with >15% settleable solids.

**Chemical Waste:** a product or by-product of an industrial process that results in a matrix not previously defined.

**Air:** whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

**Matrix Spike (spiked sample or fortified sample):**

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

**Matrix Spike Duplicate (spiked sample or fortified sample duplicate):**

A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

**Method Blank:**

A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

**Method Detection Limit:**

The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)



**National Environmental Laboratory Accreditation Conference (NELAC):**

A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

**National Environmental Laboratory Accreditation Program (NELAP):**

The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

**Negative Control:**

Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

**NELAC Standards:**

The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (NELAC)

**Performance Audit:**

The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

**Performance Based Measurement System (PBMS):**

A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

**Positive Control:**

Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

**Precision:**

The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

**Preservation:**

Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

**Proficiency Testing:**

A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]



**Proficiency Testing Program:**

The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

**Proficiency Test Sample (PT):**

A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

**Quality Assurance:**

An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

**Quality Assurance [Project] Plan (QAPP):**

A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

**Quality Control:**

The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

**Quality Control Sample:**

An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

**Quality Manual:**

A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

**Quality System:**

A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

**Quantitation Limits:**

The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

**Range:**

The difference between the minimum and the maximum of a set of values. (EPA-QAD)

**Reagent Blank (method reagent blank):**

A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

**Reference Material:**

A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

**Reference Method:**

A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

**Reference Standard:**

A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

**Replicate Analyses:**

The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

**Requirement:**

Denotes a mandatory specification; often designated by the term "shall". (NELAC)

**Resource Conservation and Recovery Act (RCRA):**

The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

**Safe Drinking Water Act (SDWA):**

The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

**Sample Duplicate:**

Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

**Second Order Polynomial Curve (Quadratic):** The 2<sup>nd</sup> order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2<sup>nd</sup> order regression will generate a coefficient of determination (COD or  $r^2$ ) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes,  $r^2$  must be greater than or equal to 0.99.

**Selectivity:**

(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

**Sensitivity:**

The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

**Spike:**

A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.. (NELAC)

**Standard:**

The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

**Standard Operating Procedures (SOPs):**

A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

**Standardized Reference Material (SRM):**

A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

**Supervisor (however named):**

The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

**Surrogate:**

A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit):

A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director:

Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test:

A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method:

An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Toxic Substances Control Act (TSCA):

The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability:

The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

Uncertainty:

A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

United States Environmental Protection Agency (EPA):

The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation:

The process of substantiating specified performance criteria. (EPA-QAD)

Verification:

Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

**Work Cell:**

A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

**Acronyms:**

BS – Blank Spike  
BSD – Blank Spike Duplicate  
CAR – Corrective Action Report  
CCV – Continuing Calibration Verification  
CF – Calibration Factor  
CFR – Code of Federal Regulations  
COC – Chain of Custody  
CRS – Change Request Form  
DOC – Demonstration of Capability  
DQO – Data Quality Objectives  
DU – Duplicate  
DUP - Duplicate  
EHS – Environment, Health and Safety  
EPA – Environmental Protection Agency  
GC - Gas Chromatography  
GC/MS - Gas Chromatography/Mass Spectrometry  
HPLC - High Performance Liquid Chromatography  
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy  
ICV – Initial Calibration Verification  
IDL – Instrument Detection Limit  
IH – Industrial Hygiene  
IS – Internal Standard  
LCS – Laboratory Control Sample  
LCSD – Laboratory Control Sample Duplicate  
LIMS – Laboratory Information Management System  
MDL – Method Detection Limit  
MS – Matrix Spike  
MSD – Matrix Spike Duplicate  
MSDS - Material Safety Data Sheet  
NELAC - National Environmental Laboratory Accreditation Conference  
NELAP - National Environmental Laboratory Accreditation Program  
PT – Performance Testing  
QAM – Quality Assurance Manual  
QA/QC – Quality Assurance / Quality Control  
QAPP – Quality Assurance Project Plan  
RF – Response Factor  
RPD – Relative Percent Difference  
RSD – Relative Standard Deviation  
SD – Standard Deviation  
SOP: Standard Operating Procedure  
TAT – Turn-Around-Time  
VOA – Volatiles  
VOC – Volatile Organic Compound

## Appendix 6.

### Laboratory Certifications, Accreditations, Validations

TestAmerica Knoxville maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Lab ID(Certificate Number)*	Organization	Lab ID(Certificate Number)*
Arkansas DEQ	88-0688	New York DOH	10781
California DHS ELAP	2423	North Carolina DENR	64
Colorado DPHE	NA	North Carolina DHHS	21705
Connecticut DPH	PH-0223	Ohio EPA VAP	CL0059
Florida DOH	E87177	Oklahoma DEQ	9415
Georgia DNR EPD	906	Pennsylvania DEP	68-00576
Hawaii DOH	NA	South Carolina DHEC	84001001
Illinois EPA	200012	Tennessee DOH	02014
Indiana DOH	C-TN-02	Tennessee DEC Radioactive Materials License	R-47060-B07
Iowa DNR	375	Utah DOH	QUAN3
Kansas DHE	E-10349	Virginia DGS	00165
Kentucky DEP	90101	West Virginia DEP	345
Kentucky DEP USTB	0078	West Virginia DHHR	9955C
Louisiana DEQ	83979	Washington DOE	C1314
Louisiana DOHH	LA070012	Wisconsin DNR	998044300
Maryland DOE	277	Navy Facilities Engineering Service Center	NA
Massachusetts DEP	M-TN009	US Department of Agriculture	S-46424
Michigan DEQ	9933	US Department of Energy	NA
New Jersey DEP	TN001	US Army Corps of Engineers	NA

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, and on the local server in a protected QA share.

\* The Agency assigned Laboratory ID number is listed where provided. Otherwise the number corresponds to the Agency Certificate ID where provided.

## Appendix 7. Example Data Qualifiers (Non-Isotope Dilution Methods)<sup>1</sup>

Qualifier	Group	Footnote
D	ALL	Result was obtained from the analysis of a dilution.
DIL	ALL	The concentration is estimated or not reported due to dilution or the presence of interfering analytes.
G	ALL	Elevated reporting limit. The reporting limit is elevated due to matrix interference.
MSB	ALL	The recovery and RPD were not calculated because the sample amount was greater than four times the spike amount.
NC	ALL	The recovery and/or RPD were not calculated.
ND	ALL	Analyte analyzed for but was not detected.
NR	ALL	Not reportable.
AP	GC/HPLC	Altered Pattern
COL	GC/HPLC	More than 40% difference between primary and confirmation column results. The lower of the two results is reported.
PE	GC/HPLC	The %Difference between the primary and confirmation columns exceeds 40%. The higher value is reported.
N	GCMS	Estimated result. Analyte is a Tentatively Identified Compound (TIC).
*	Inorganics	Relative percent difference (RPD) is outside stated control limits.
B	Inorganics	Estimated result. Result is less than RL.
E	Inorganics	Matrix interference.
J	Inorganics	Method blank contamination. The associated method blank contains the target analyte at a reportable level.
N	Inorganics	Spiked analyte recovery is outside stated control limits.
*	Organics	Surrogate recovery is outside stated control limits.
A	Organics	Spiked analyte recovery is outside stated control limits.
B	Organics	Method blank contamination. The associated method blank contains the target analyte at a reportable level.
E	Organics	Estimated result. Result concentration exceeds the calibration range.
I	Organics	Matrix interference.
J	Organics	Estimated result. Result is less than RL.
P	Organics	Relative percent difference (RPD) is outside stated control limits.
HC	WET	Initial dilution due to High Conductivity

1 – This is a standard list of qualifiers commonly used. Other qualifiers are available in LIMS.



## Appendix 7. Continued Example Data Qualifiers (Isotope Dilution Methods)<sup>1</sup>

Qualifier	Group	Footnote/Description
J	Dioxin/ Specialty Organics	The reported result is an estimate. The amount reported is below the Minimum Level (ML). The qualitative definition of the ML is "the lowest level at which the analytical system must give a reliable signal and an acceptable calibration point". The ML was introduced in EPA Methods 1624 and 1625 in 1980 and was promulgated in these methods in 1984 at 40 CFR Part 136, Appendix A. For the purposes of this report the ML is qualitatively defined as described above, and quantitatively defined as follows: <b>Minimum Level:</b> The concentration or mass of analyte in the sample that corresponds to the lowest calibration level in the initial calibration. It represents a concentration (in the sample extract) equivalent to that of the lowest calibration standard, after corrections for method-specified sample weights, volumes and cleanup procedures has been employed.
E	Dioxin/ Specialty Organics	The reported result is an estimate. The amount reported is above the UCL described below. The E qualifier is applied on the basis of the Upper Calibration Level (UCL). The quantitative definition of the UCL is listed below: Upper Calibration Level: The concentration or mass of analyte in the sample that corresponds to the highest calibration level in the initial calibration. It is equivalent to the concentration of the highest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.
B	Dioxin/ Specialty Organics	The analyte is present in the associated method blank at a reportable level. For this analysis, there is no method specified reporting level, other than the qualitative criterion that peaks must exhibit a signal-to-noise ratio of 2.5-to-1. Therefore, the presence of any amount of the analyte present in the blank will result a B qualifier on all associated samples. If the blank has analytes present above the ML (described above) the need for corrective action beyond qualifying the associated data is evaluated. The determination is made whether the amount in the blank is less than 5% of the lowest amount in associated client samples or regulatory limit. If this is the case, sample processing may continue with the qualification of the data. If the amount in the blank is greater than 5% of the lowest amount in associated client samples or regulatory limit, corrective action must be taken. The corrective actions may include extracting a second aliquot of sample if available, or notifying the client to assess the impact on the project objectives.
Q	Dioxin/ Specialty Organics	Estimated maximum possible concentration. This qualifier is used when the result is generated from chromatographic data that does not meet all the qualitative criteria for a positive identification given in the method. The criteria include the following areas: <ul style="list-style-type: none"> <li>• Ion abundance ratios must be within specified limits (+/-15% of theoretical ion abundance ratio.)</li> <li>• Retention time criteria (relative to the method-specified isotope labeled retention time standard).</li> <li>• Co-maximization criterion. The two quantitation ion peaks must reach their maxima within 2 seconds of each other.</li> <li>• Polychlorinated dibenzofuran purity. No peak can be identified as a polychlorinated dibenzofuran if a polychlorinated diphenyl ether peak maximizes within +/- 2 seconds of the furan candidate.</li> </ul>
S	Dioxin/ Specialty Organics	Ion suppression evident. The trace indicating the signal from the lock mass of the calibration compound shows a deflection at the retention time of the analyte. This may indicate a temporary suppression of the instrument sensitivity, due to a matrix-borne interference.
C	Dioxin/ Specialty Organics	Coeluting Isomer. The isomer is known to coelute with another member of its homologue group, or the peak shape is shouldered, indicating the likelihood of a coeluting isomer
X	Dioxin/ Specialty Organics	Other. See explanation in narrative

<sup>1</sup> - This is a standard list of qualifiers commonly used. Other qualifiers are available in LIMS